Chemical & Process Engineering

Vol. 36, No. 3 | MARCH 1955

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***Subscription rates:** 1 year 8 dollars, 3 years 20 dollars, paid in advance*

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Chemical Engineers and Centipedes

THE question 'What is a chemical engineer?' has been posed with increasing frequency during the past decade or so and a variety of answers, right or wrong, have been forthcoming. Sometimes the questioner is subjected to a rapid course in unit operations. Sometimes it is explained how it all started from co-operation between chemist and engineer, or a picturesque account is given of the scaling-up of laboratory apparatus until it becomes chemical plant. Or, more boldly, a concise definition is attempted.

The last method is the most appropriate, if the most difficult, in these times. The chemical engineer is now employed in so many industries, in so many different ways, that the older conception of a chemical engineer—one might be pardoned for imagining him as having G. E. Davis' handbook for his 'bible' and a string of unit operations as his 'rosary'—is no longer valid.

This month an important conference, whose functions we described last month, is being held in London. Under the chairmanship of that eminent British expert, Sir Harold Hartley, K.C.V.O., F.R.S., well-known authorities from many parts of Europe will discuss topics relating to the provision of chemical engineering education and research facilities. The conference will also draw attention to the need for a wider recognition of chemical engineering's vital role in modern industry.

Let us hope that there will also emerge from this conference a more clear-cut and up-to-date answer to the question, 'What is a chemical engineer?'

Whether or not this conference gets the wide publicity it deserves, it is likely to stimulate discussion, in many quarters, on the nature of chemical engineering and the functions of the chemical engineer. This raises the question, how far should the ordinary chemical engineer himself be concerned with such discussion? For, granted that it is essential for the precise boundaries of chemical engineering to be clearly appreciated—for instance, by industrial managers who have to co-ordinate chemical engineers in their organisation, and by teachers of chemical engineering in universities and colleges—too much preoccupation with the question by working chemical engineers might be harmful.

Somehow, there springs to mind the old story about the centipede who was approached by an inquisitive person one day and asked which of his many legs, when he moved, he moved first. The centipede was rooted to the spot for days afterwards.

As in any other profession, the chemical engineer is given a certain amount of knowledge and taught, in a broad way, how to apply it. As he moves around in industry he acquires what is called 'experience.' But the chemical engineer's lot is a particularly hard one as, with the rapidly changing pattern of industry today,

with new technologies and new materials being created, he comes against problems for which his college teachers, unless gifted with second sight, could have given him scant preparation. His final asset, the ultimate proof of his competence, is that 'flair,' that instinct, which defies description or analysis but which enables him to overcome any genuine chemical engineering problem as surely as any centipede can walk.

Fibres in industry

'NYLON' has become synonymous with 'stocking' to women today and, even to those of us who keep step with the march of technology, it comes as a shock to realise that it is only just over 15 years ago that this—the first truly synthetic fibre—was placed on the market by the Du Pont Co. The product of seven years of painstaking research, nylon was first produced on a full scale only at a cost of $27 million in investment and the cumulative efforts of some 230 chemists and engineers.

As nylon goes into its sixteenth year, it carries no light burden of duties. Many of its uses are on the side of gaiety: sheer hosiery and dainty garments for women; nylon shirts and socks for men. But a large percentage of production goes to work for industry: filter cloths, bolting cloths for the food industry, protective clothing, drive belts, conveyor belts and gaskets spring to mind as well as tyres and ropes.

Imperial Chemical Industries' new *Terylene* plant at Wilton, featured on another page, brings into prominence another fibre with great possibilities in the industrial field. The high resistance of *Terylene* to heat and chemicals has made it useful in filtration processes involved in the manufacture of dyestuffs and phosphates and in other industries. The properties of the fibre which relate to its use in filtration processes, coupled with a very low moisture absorbency and resistance to flexing, also make it useful to the paper-making industry. Felts for the processing of paper have been produced and successfully tested. Also being developed from *Terylene* is industrial clothing designed to give protection against acids, and encouraging results of tests under working conditions are already available. These are only a few of the polyester fibre's industrial possibilities and it is already making a significant contribution in laundering and dyeing.

Glass fibres, with their high tensile strength, have provided a new field for the use of fibres in industry. Offering not only high strength and low weight, but also chemical inertness and ease of fabrication, glass-reinforced plastics have already made their mark in the motor-car, boat-building, electrical and certain other industries, and interest in their chemical engineering possibilities is increasing. One attractive feature is that they can be fabricated into equipment such as tanks, trays and ducting, etc., with inexpensive moulds without the application of heat or pressure. Undoubtedly we shall hear much more of this material in the future.

New chemical engineering uses for PTFE

UNIQUE among organic compounds in its chemical inertness, its toughness over a wide temperature range and other properties, polytetrafluorethylene (PTFE) is being put to a number of uses by the chemical engineer. Difficulties of processing and fabricating this material have hindered its wider application in the past; manufacturers, however, are fast finding ways of getting round these problems. Thus, Crane Packing Ltd. have now found a way of extruding PTFE rod and tube up to an external diameter of 4 in. Tube of this diameter has a minimum wall thickness of ¼ in. The standard length of both tube and rod turned out by Crane is 3 ft.

Users of PTFE will be aware of its 'memory characteristics' and of the tendency of fabrications to return, in certain circumstances, to previous basic forms. It is stated that, in the new extruded technique, this phenomenon is eliminated.

Another British concern, the Edison Swan Electric Co. Ltd., is now able to produce PTFE bonded to metal or rubber, PTFE-bonded fibre-glass laminates, continuous-length cylinders and laboratory beakers of PTFE. In the metallising process, it would seem that bonding takes place during manufacture of the PTFE, the surface layers being penetrated by microscopic filaments from the metal to make a firm bond. Although brass has been chiefly used up to now, success has also been achieved with zinc, copper and other non-ferrous metals, and ferrous metals seem a possibility to be explored.

A laminate of PTFE bonded with rubber, combining the chemical inertness of the one with the flexibility of the other, is useful for valve and pump diaphragms, washers, seals and flexible couplings, while continuous cylinders of PTFE are used in the food processing and other industries for covering rollers handling sticky materials.

Russian potash expansion

A SCHEME for an expansion of the potash industry in Russia has been reported recently. The Russians probably possess the richest potassium salt deposits in the world, but, although these were already discovered in 1916, no salts were mined until 1934 because of the remoteness of these deposits on the western slopes of the Ural mountains far away from the coast—1,200 miles from Leningrad and 1,300 miles from the Black Sea—and far away from the agricultural districts in need of potash fertilisers.

The composition of the Ural deposits is similar to that of the famous German potassium salt deposits, of which 60% are now under the control of the Russians. The main mineral is carnallite, a hydrated potassium magnesium chloride with a potassium content equivalent to 12% K_2O. The lower layers consist of sylvinite, a mixture of potassium and sodium chlorides with a potassium content equivalent to 14 to 25% K_2O.

According to an article in *Chemische Industrie* of November 1954, the Russians have founded a new potash combine in the neighbourhood of the town of

Comical Engineering Terms

"SINK AND FLOAT CONCENTRATION"

Bjerjesniki, situated on the river Kama. Mining commenced last autumn. Modern methods are used to raise the sylvinite from a depth of 1,000 ft., and conveyor belts then transfer the mineral into 5-ton trucks. These are drawn by electric locomotives to the works, where the crude salt is ground and recrystallised under reduced pressure. The annual output is expected to reach some hundred thousand tons of potash fertilisers.

Chemical works safety

THE switch, this year, from the theoretical to the practical at the A.B.C.M. Chemical Works' Safety Conference—by organising works visits instead of the usual presentation and discussion of papers—seems to have been a successful stroke of policy well received by the 380 delegates who attended. Apart from the obvious attraction of an opportunity to tour Imperial Chemical Industries' big works at Billingham and Wilton, this sort of close contact with the subject is much more conducive to a live discussion than any paper, however well prepared and presented. Inspection of the I.C.I. safety methods led to some interesting points being brought out at the 'quiz' session, held subsequently. Indeed, more questions were submitted than could be dealt with in the time available and, in addition, some further questions were received later.

All these questions, and the answers that have been given to them, have now been recorded in the proceedings of the conference, which has now been issued by the Association of British Chemical Manufacturers (price 3s.). This 48-page booklet starts off with a brief introduction which is followed by the introductory addresses that were made to the delegates by Dr. I. J. Faulkner, of Billingham, and Dr. C. Cockram, of Wilton. The next two sections contain descriptive outlines of the visits to Billingham and Wilton, respectively, and finally there are the delegates' questions and the answers given to them. The booklet will serve as a useful record of a noteworthy conference.

Boiler feedwater treatment at 2,000 gal./min.

THE problem of boiler feedwater treatment, discussed in an article in this issue, becomes staggering in a power plant of central station proportions that operates with 100% make-up. The steam generating unit at Dow Chemical Co.'s Midland, Mich., plant operates under just these conditions and for this reason must be considered unique. One of its steam generators turns out 400,000 lb. of 1,250-p.s.i. steam every hour. Since the steam is used for chemical processing after passing through the turbines, all the boiler feed must be made up from raw water. This means water treatment on an unprecedented scale—800 gal. demineralised every minute for each generator. When two other boilers are completed, the demineralising units will operate at their full capacity of 2,000 gal./min.

At Midland, demineralisation is accomplished by massive cation and anion exchangers. This equipment uses the cation and anion remover process with silica removal. The treatment is so effective that the demineralised water consistently shows an average silica content of less than 0.2 p.p.m. More than half the run shows less than 0.1 p.p.m. silica. In a second demineralising plant, now under construction at Midland, silica will be removed by direct absorption into the anion resin.

As a last step, carbon dioxide and other dissolved gases are almost completely removed in a vacuum deaerator. The CO_2 in the effluent deaerator is less than 2 p.p.m., and oxygen is less than 0.1 p.p.m.

The demineralising plant has been described in a technical bulletin of Henry Wiggin & Co. Ltd. The extensive use of *Monel* for handling the various corrosive chemicals is also referred to.

Israel's new uranium and heavy water processes

IMPORTANT discoveries have been made by atomic scientists in Israel, but some exaggerated accounts have appeared concerning them. A recent broadcast by Dr. Ernst D. Bergmann, chairman of the Israeli Atomic Energy Commission, puts the matter in perspective. Israel's work in this field rests chiefly on two facts—that some of the minerals found in the Negev, especially the phosphates, contained uranium, and that at the time this discovery was made the Weizmann Institute of Science was studying a method for separation of isotopes which appeared to be applicable to the production of heavy water from ordinary water.

Israeli atomic scientists have therefore two main tasks: to study the possibilities of exploiting the

radioactive ores and of the industrial production of uranium—and to develop, on the strength of the results, previously achieved, a process for the production of heavy water.

In the field of heavy water, further development work has been carried out on the method for the enrichment of heavy water which does not require electric energy; this investigation is still continuing. Results on a laboratory scale give reason to hope that, on an industrial scale also, this method will be able to compete with the electrolytic method. However, it is yet too early for a definite statement on this subject.

In the field of uranium, the results achieved are somewhat more advanced. A method has been worked out for the production of uranium from phosphates, and this method has been tested on a pilot-plant scale. It is true that the uranium content of the phosphates is low, but it is known already that the cost of uranium produced by this method is commensurate with the price of that metal, when produced by the usual methods from rich ores.

There is, therefore, a good chance that the State of Israel will be able to produce uranium on an industrial scale in the not-too-distant future.

This discovery appears to be of more general importance. The world possesses a finite quantity of rich uranium minerals and very large quantities of poor ores. To utilise the energy contained in the radioactive elements for constructive purposes, we shall have to find a method for the exploitation even of the low-grade ores, and it appears that the new method is applicable not only to the phosphates found in Israel.

New chipboard process

CHIPBOARD manufacture in the United Kingdom so far has usually been carried out by the consolidation under heat and pressure of wood chips or other industrial wood waste to which has been added a thermosetting resin adhesive. A new factory in Scotland has been designed to make use of specially prepared wood chips whose dimensions are carefully controlled, and which are prepared from essentially one species of softwood. This has the advantage of enabling the manufacturer to produce chipboard designed for specific purposes. Another departure from current procedure is that, instead of the conventional uniform board, the new type chipboard will make use of a highly efficient method of chip grading which ensures that the fine material remains at the surface of the board.

The basic raw material is forest thinnings, which are converted to chips of the correct size and moisture content and then conveyed to specially designed rotary mixers. On passing through the mixers they are sprayed with a synthetic resin adhesive mix in accurately controlled proportions by weight. The mixture of chips and resin is spread on aluminium trays in which the boards are formed. Unlike existing processes, it is necessary to be able to handle the mass of chips, or mattress as it is called; pre-pressing technique with very careful moisture control is introduced for this purpose. The press used is a multi-platen, steam-heated hydraulic press and the cycle of operations is carefully arranged to ensure that there is no possibility of untimely curing of the synthetic resin.

The resin used is urea formaldehyde with a shelf life of several months. It is used in conjunction with a hardener specially developed to overcome various faults previously experienced, such as residual formaldehyde in the boards.

The business of mixing the resin is not such a simple matter as might be thought. The ideal is to have a thin, even coating on the surfaces of the wood chips. To approach this requires a knowledge of the species of wood used, the manner in which it has been cut, *i.e.* along or across the grain, and a knowledge of the spray nozzle characteristics for different viscosities and pressures. The dimensions of the mixing chamber and the time of transit of the chips through the mixer are also of primary importance in considering the efficiency and economics of the mixing stage.

The new factory is that of the Airscrew Co. & Jicwood Ltd. at Annan in Dumfriesshire.

Illustrated Pre-View of the Achema XI Congress and Exhibition

The Achema XI Chemical Plant and Apparatus Exhibition and Congress will be held at Frankfurt-am-Main, Germany, from May 14 *to* 22, 1955, *in connection with the* 1955 *Congress of the European Federation of Chemical Engineering. The Achema XI is organised by Dechema (Deutsche Gesellschaft für Chemisches Apparateswesen), the German association of chemical plant manufacturers.*

The Achema exhibition is recognised as an important international event, providing a means of bringing together chemists, engineers and others to inspect and discuss new developments in plant, apparatus, instruments, constructional materials and other products for use in the chemical and associated industries.

The April issue of CHEMICAL & PROCESS ENGINEERING *will contain a special section devoted to the Achema XI and including an illustrated pre-view of the many new items of plant, apparatus and products that will be exhibited.*

MECHANICAL SEALS

A Survey of Present-Day Practice—Part I

By B. G. Williams, A.C.G.I., B.SC.(ENG.), A.M.I.MECH.E., A.M.I.CHEM.E.

Mechanical seals have come into prominence in recent years as a solution to the shaft sealing problem in many pumping installations. The author reviews the whole question of mechanical seals in an article which will be presented in two parts. In this first part he discusses how and when to use mechanical seals, different types available, materials of construction, design considerations, and the effect of pump design.

ALTHOUGH mechanical seals were in service in refineries in the U.S.A. prior to 1939, their full development was delayed by World War 2, but since 1947 the field of application in this country has expanded at a phenomenal rate.

The urgent demand in the oil industry provided the necessary impetus for the development of mechanical seals to supply the solution to many difficult stuffing-box problems, particularly when handling light hydrocarbon fractions under saturation pressure and temperature conditions.

A typical example is propane, which has a saturation vapour pressure of 300 p.s.i.a. at 140°F. Pumps operating on this duty previously needed an elaborate sealing system. The seal oil was circulated through the lantern ring of the pump stuffing box at a pressure slightly below the product pressure (in order to prevent degradation of product). Careful design of the stuffing box was essential for the following reasons:

(*a*) The maintenance of a constant steady pressure, under accurate control, in the pump stuffing-box lantern ring.

(*b*) Continuous circulation of sealing oil.

(*c*) Adequate supply of seal oil.

(*d*) Circulation of seal oil through the stuffing box once only to prevent dilution.

The single mechanical seal has been developed in order to eliminate this elaborate sealing system.

With conventional-type soft packings a certain amount of leakage is a desirable feature, in order to provide lubrication and cooling for the packings. As a general rule, the tighter packings are maintained to minimise gland leakage the higher are the maintenance costs due to sleeve scoring and packing replacement.

In certain circumstances a reasonable amount of leakage can be tolerated,

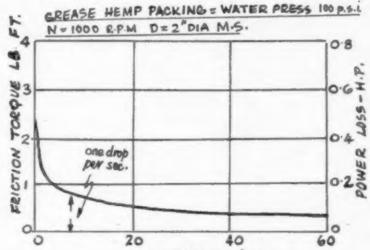


Fig. 1. Leakage-friction torque diagram.

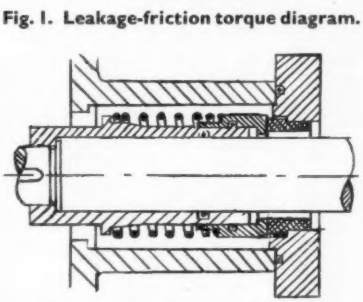


Fig. 2. Typical mechanical seal.

as in the case of water pumps and when handling the heavier petroleum products which can be recovered at nominal cost and where there is no appreciable loss or degradation of the stock. Under these service conditions long packing life is generally experienced, and at first glance the installation of mechanical seals may be hard to justify.

On chemical work, certain important inherent properties of the chemical products must be considered. Many acids may be relatively inert inside the pump away from the contact of air and moisture. They may even be excellent lubricants, as in the case of concentrated sulphuric acid and liquid sulphur dioxide. When leakage of acid occurs through the stuffing box the rate of corrosion is immediately accelerated by contact of the acid with oxygen and moisture in the atmosphere.

At pressures below atmospheric the entry of moisture and oxygen may lead to serious corrosion not only of the pump but the whole pumping system.

At temperatures below atmospheric, insidious leakages which cannot normally be detected by hydraulic pressure tests, together with the increased damp conditions due to the condensation of atmospheric moisture, may lead to rapid chemical attack on the pump from the outside.

In some plants, discouraging initial experiences have resulted in the banning of mechanical seals. On the other hand, many other users have used mechanical seals with increasing success. As is true of almost all new methods, many of the failures are the result of operators and engineers who do not understand the problems of the new equipment as well as that which they used since their apprenticeship.

Conventional packed glands

It has already been mentioned that with conventionally packed glands it is necessary to allow a small leakage of product to provide lubrication and cooling.

Under these conditions a thin film of product separates the shaft and packing and prevents excessive friction. This film arises from the slightly out-of-true rotation of the pump shaft.

When the packing is compressed tightly so that leakage is prevented, the intense frictional heat generated leads to expansion of the shaft and packing, so that the shaft becomes grooved and burnt and the packing destroyed.

Experiments[1] have shown that the commonly held idea that grease-filled packing is self-lubricating is untrue.

Fig. 1 shows the effect on friction of tightening a grease-filled hemp-packed gland. When the leakage is reduced below one drop per second there is a large increase in the frictional heat generated. After tightening

the gland it may be many hours before the full effect is noticed. Leakage may even temporarily increase after tightening.

Metal or synthetic rubber packing rings normally used in the petroleum industry as grease-filled packings are rapidly degreased by light fractions and solvents.

Typical mechanical seals

A typical mechanical seal is shown in Fig. 2. The stationary element of the seal consists of the gland or seal plate, its retaining assembly and the gland insert. The gland may be made of mild steel, except where corrosion dictates the use of alloys. It is essential that the seal gland is designed with sufficient thickness to prevent distortion when it is bolted up.

The principle of mechanical sealing is based on the intimate contact of two perfectly flat faces—one stationary and the other rotating. Consequently, no deformation of the gland can be tolerated, as the gland insert, if mounted with a press fit, would suffer the same amount of deflection and cause failure.

The joint between the gland and pump body would also be affected. The thickness of the conventional stuffing-box gland is not as critical as the thickness of the seal gland, since the force required is only necessary to hold the soft packing in the stuffing box against the hydrostatic pressure.

The seal gland must not only resist the hydrostatic pressure but also compress and seat the joint used, thereby requiring greater strength and consequently increased thickness. Since the force required to retain seal glands is many times that needed for soft packings, seal gland bolts must be carefully designed.

Gland-bolt design is generally based on paragraph W-317 of the 1951 edition of the A.P.I.-A.S.M.E. code for unfired pressure vessels. A design stress of 30,000 p.s.i. has been used by some designers[2] instead of the 20,000 p.s.i. specified by the code, since the seal glands are not subjected to bending moments.

In many cases on existing pumps three or four $\frac{1}{32}$-in.-thick composition gland gaskets are used each time a seal is assembled, to provide a quick adjustment for seal face wear.

Up to stuffing-box pressures of 400 p.s.i. the force required to seat the seal gland gasket governs gland-bolt design, but above 400 p.s.i. the fluid pressure governs bolt working load determinations.

Seal glands are often provided with auxiliary packing glands with the idea of controlling complete seal failures. The ordinary non-lubricated type of auxiliary packing is not satisfactory in controlling leakage, particularly with light hydrocarbons. The two or three rings of soft packing, continuously running dry, rapidly lose their effectiveness.

Single and double seals

For many years the trend was towards the replacement of soft packings with double mechanical seals using seal oil. In more recent years the trend has been the replacement of double mechanical seals with single mechanical seals which do not require separate sealing systems.

The use of double mechanical seals employing seal oil, however, is often warranted and is still standard practice in some refineries for sealing against high-temperature oil streams and toxic liquors such as acid-treated oils, phenol solutions, acids and other chemicals.

Double mechanical seals, as the name implies, consist of two single seals placed back to back in the same stuffing box, as shown in Fig. 3. Flushing liquid must be supplied to the space between the seals at a sufficient pressure to keep the seal faces in contact and to provide lubrication and cooling.

It may be safely said that anything may be successfully sealed with double seals. The disadvantage in using double seals lies in the fact that an auxiliary flushing system is necessary. The possibility of product degradation also exists, due to the possibility of seal oil leaking into the pump through the inside seal when flushing oil and product pressures fluctuate.

Balanced and unbalanced seals

Mechanical seals may be designed as balanced or unbalanced. These

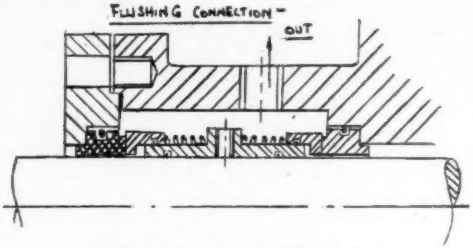


Fig. 3. Typical double seal.

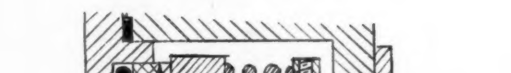


Fig. 4. Unbalanced, partially-balanced and fully-balanced seals.

terms refer to the design of the seals in so much as the hydraulic forces affect the tightness of the sliding contact between the sealing forces.

On an unbalanced seal the force resulting from the pressure on the rotating ring is carried by the seal faces acting as a thrust bearing. An increase in the stuffing-box pressure will be accompanied by a greater thrust of the rotating seal ring on the carbon insert. Large resultant forces in unbalanced seals cause rapid wear and large frictional heat generation which may lead to failure. Consequently, the change from the cheaper unbalanced seal to the more expensive balanced types generally occurs at sealing pressure between 75 and 150 p.s.i.

Also, when pumping light hydrocarbons where relatively high suction pressures are used and on applications where poor lubricating properties exist with the products being handled, it is highly desirable to used balanced seals.

On a fully balance seal, the opposing pressures which are applied to the seal are equalised and the thrust on the seal faces remains constant and independent of stuffing-box pressures. The equalising of the opposing pressures may be achieved either by means of a stepped shaft or the use of a sleeve anchored to the shaft. In both cases the unbalancing thrust on the sleeve is transmitted to the pump thrust bearings.

The degree of balance of the seal may be calculated by the following formula:

$$\% \text{ balance} = 100\left(1 - \frac{D_2^2 - D_3^2}{D_2^2 - D_1^2}\right) \quad .. (1)$$

where D_1 = i.d. of carbon ring, D_2 = o.d. of carbon ring and D_3 = o.d. of sleeve (Fig. 4).

Let the stuffing-box pressure be P p.s.i. and the average carbon seal face pressure be P_f p.s.i., then

$$P\frac{\pi}{4}(D_2^2 - D_3^2) = P_f\frac{\pi}{4}(D_2^2 - D_1^2) \quad .. (2)$$

$$P_f = P\left(\frac{D_2^2 - D_3^2}{D_2^2 - D_1^2}\right) \quad ...... (3)$$

$$\text{degree of balance} = \left(1 - \frac{P_f}{P}\right) 100$$

$$\text{or} = 100\left(1 - \frac{D_2^2 - D_3^2}{D_2^2 - D_1^2}\right) \quad ...... (1)$$

Typical unbalanced and partially balanced seals are shown in Figs. 5 and 6 respectively.

Fully balanced seals are not generally practical and some degree of unbalance is usually included in the design to provide a positive closing force as the pump discharge pressure increases to prevent the seal opening on a pressure surge.

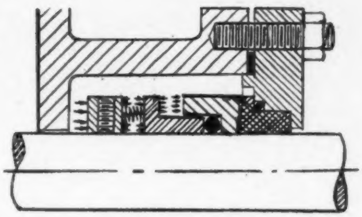


Fig. 5. Typical unbalanced seal.

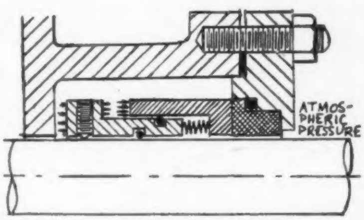


Fig. 6. Typical partially-balanced seal.

Many users standardise partially balanced seals in order to eliminate the possibility of misapplication when a pump is transferred to a different operation with high suction pressures.

Seal face pressure

Let us call the average seal face pressure P_f. Whilst the pressure drop across the faces is not a straight-line relationship, it will be assumed as such for the purpose of illustration, in which case the average seal face pressure will be $\frac{P}{2}$ above atmospheric pressure, where P is the pressure in the stuffing box.

In the unbalanced seal shown in Fig. 7 (*a*)

$$P_f \times A_1 = P \times A_2 + P_s$$

$$P_f = \frac{PA_2}{A_1} + \frac{P_s}{A_1}$$

However, $A_2 > A_1$. Consequently $P_f > P$ and the faces will run dry. This will cause the faces to become hot owing to the frictional heat generated. It will be seen that with this type of seal good product circulation in the gland should be provided to dissipate the heat generated and to prevent local increases in vapour pressure.

With partially and fully balanced seals P_f is less than P (Fig. 7 (*b*)) and liquid can find its way between the seal faces, with the result that the pressure drops from P to atmospheric pressure across the faces. The slight leak of liquid between the seal faces provides the necessary lubrication to render seal face wear unmeasurable and, as this leak is less than the evaporation rate, no visible leakage can be observed.

(In Fig. 7 (*b*), $P_f \times A_1 = P \times A_2 + P_s$. In order that seal faces are kept closed by pressure of spring only, then $P_f \times A_1 = P \times A_2$. But $P_f = \frac{P}{2}$, therefore A_1 must be equal to $2A_2$.)

If there is any difficulty in visualising the mechanism by which the fluid initially penetrates between the seal faces, it must be remembered that the product will find its way into the inaccuracies even between two optically flat surfaces and, of course, when the pump is operating—for example at 2,950 r.p.m.—the seal must flex 2,950 times per minute. Even the slightest delay in response will allow product to enter between the seal faces.

Partially balanced seals must inevitably give longer average lives than unbalanced seals. The extent of the partial balancing, when referred to an existing pump, is limited by the area of the stuffing box.

Maximum practical degree of balance

Referring again to Fig. 7 (*b*), it is seen that the maximum practical degree of balance is achieved when $A_1 = 2A_2$, *i.e.* $D_2^2 - D_1^2 = 2(D_2^2 - D_3^2)$

$$\text{or } \frac{D_2^2 - D_3^2}{D_2^2 - D_1^2} = \tfrac{1}{2}$$

The degree of balance may then be found by substitution in equation (1).

$$\begin{aligned} \% \text{ balance} &= 100\left(1 - \frac{D_2^2 - D_3^2}{D_2^2 - D_1^2}\right) \\ &= 100\,(1 - \tfrac{1}{2}) \\ &= 50\% \end{aligned}$$

When a seal is theoretically 100% balanced, then $D_2 = D_3$ and the seal will take the form as shown in Fig. 7 (*c*). It will be seen that the seal is 100% balanced with regard to the stuffing-box fluid pressure, but the seal face pressure $P_f\left(=\frac{P}{2}\right)$ must inevitably open the seal faces, and consequently the design is not a practical proposition.

Sleeve and seal ring

The sleeve and seal ring (Fig. 2) are generally made of stainless steel or, when the duty demands, a more

expensive corrosion-resistant hard material.

The sleeve performs the dual task of seating the shaft O-ring and driving the seal ring.

Clearance between the shaft and sleeve is usually held to .010 to .012 in. for best results.

In some modern designs the sleeve is replaced by a collar which is anchored to the shaft by means of an Allen screw (Fig. 9). The clearance between the shaft and collar is usually between .002 and .003 in. to prevent appreciable unbalance of the rotating mass of the collar.

The drive pins and compression springs (Fig. 9) are made of the same materials as the other components of the rotating element. The pins transmit the driving force from the collar to the seal or compression ring. It has been found that in certain seal designs when the springs surrounded the pins, interlocking of the collar and compression ring occurred. Those pins are now separate and are inserted in a hole located mid-distant between the springs.

Materials of construction

Gland inserts are usually made of metal-filled carbon. Originally plain carbon was employed, but its inherent porosity allowed leakage of the lighter hydrocarbon fractions. In order to overcome this difficulty, inserts filled with cadmium, lead, copper, etc., have been developed.

Worn carbon inserts, after being faced up on a lathe with a normal lathe tool, may be lapped on clean cast iron using no abrasive, as it has been found that abrasive grit will become embedded in the metal filling. Any grit contamination of the carbon face will lead to rapid failure of the seal and the scoring of the hard face of the seal ring.

In order to take every precaution that a refaced carbon insert will not blow when starting up, it is a useful tip to machine a 3° chamfer on the face, as shown in Fig. 8.

The insert will quickly wear through to its full face, but initially a high seal face pressure will be induced, preventing leakage.

(In Fig. 8, assume $A_1 = 4$ sq. in.; $A_2 = 2$ sq. in.; $A_3 = 2$ sq. in.; and $P_1 = 100$ p.s.i.

Closing force

$$= + F = P_1 \times A_3 = 200 \text{ lb.}$$

Opening force

$$= \frac{P_1 + O}{2} \times A_2 = 100 \text{ lb.}$$

Resultant closing force until face wears smooth is 100 lb. above spring pressure.)

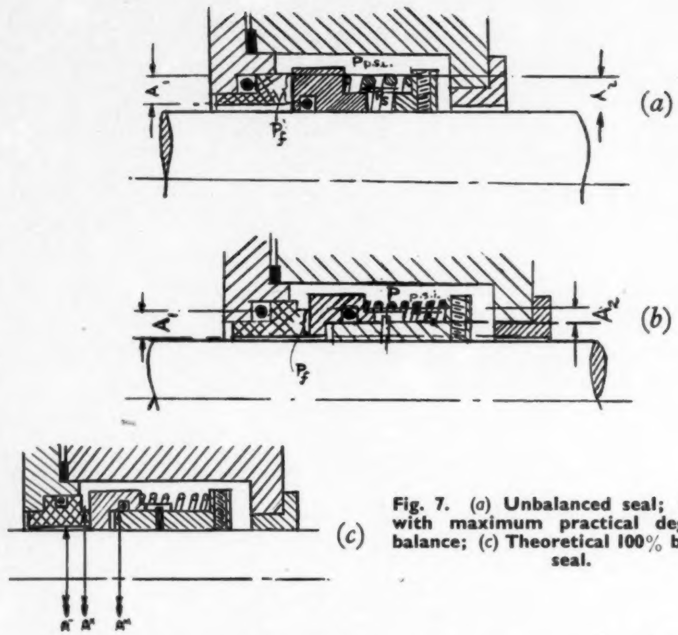


Fig. 7. (a) Unbalanced seal; (b) Seal with maximum practical degree of balance; (c) Theoretical 100% balanced seal.

Bronze has been the most successful substitute for carbon. Other experimental materials which have been employed as inserts include tungsten carbide, cast iron and thermo-plastics. Glass-filled and graphite-filled tetra-fluoro-ethylene resin stationary inserts have not proved entirely satisfactory due to excessive wear and deformation.

Seal inserts are either pressed into the gland (with a .001 to .002 in. interference fit) or mounted on O-rings. The O-ring mounting is generally preferred, although press-fitted carbons give satisfactory service.

The best all-round material for the rotating elements has proved to be the 18: 8: 1 type stainless steel. A 16½%-Cr, 2½%-Ni, stainless-steel type of seal is also marketed, being the most corrosion-resistant heat-treatable stainless steel available. This is quite satisfactory on hydrocarbon service. Plain carbon steel is generally unsatisfactory, since the slightest corrosion affects adversely the free floating movement of the seal parts.

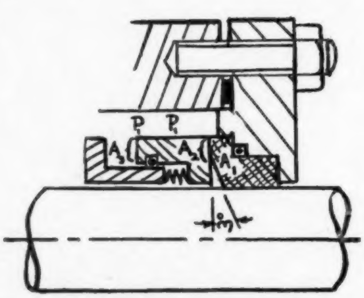


Fig. 8. Effect of chamfered carbon insert.

It is essential that the seal ring has a very hard face of *Stellite* No. 1, *Colmonoy* No. 6, tungsten carbide or other similar hard corrosion-resistant metals. These materials have characteristically high Brinell numbers of over 600 and can be readily applied to the seal-ring face by modern techniques.

The use of hardened stainless-steel seal faces has not been successful, since after hardening some of the stainless steels lose their corrosion-resistant properties. The life of such a component is also short, owing to the limited depth of the hard face.

Ceramic materials are being developed and give every indication of great promise. Applications on weak acid pumps have given excellent results. This is due to their inherent corrosion resistance and their hardness. Although naturally fragile, ceramic rings are sturdy enough for many low-pressure applications. If it is at all possible to look into the future, then without doubt ceramic seal rings are still in their infancy and future development will bring the full benefit of this corrosion- and heat-resistant hard material.

O-rings

O-rings are generally made in three basic materials: (a) synthetic and natural rubbers; (b) thermo-plastic

resins; and (*c*) composition materials such as synthetic rubber impregnated with asbestos. These O-rings are designed to be fitted with a diametral compression of between .012 and .030 in. and then, as the hydrostatic pressure is applied to either side, the O-ring grips the faces, compressing it with a tightness proportional to the applied pressure. Consequently the higher the fluid pressure applied, the tighter the seal becomes. The extent of the clearance between the shaft and the sleeve limits the pressure against which the O-ring will seal. If this clearance (which is normally held to between .010 and .012%) becomes excessive, then the higher pressures will cause the rubber O-ring to fail by extrusion through the sleeve clearance.

The effect of temperature and chemical attack must be carefully considered. With synthetic rubbers the upper temperature limit is about 200°F. and the lower limit is about -30°F. Experience has shown that acids, alkalis and hydrocarbons with high aromatic content cause rapid

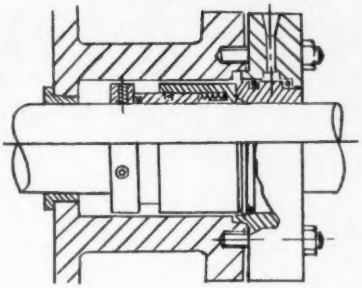


Fig. 9. Typical collar-driven seal.

deterioration of most synthetic rubbers.

When the use of synthetic rubber materials is excluded by virtue of temperature conditions or chemical attack, the thermoplastic resins may be used to extend the limits of the mechanical seal application.

These plastic materials may be used between temperature limits of 450 and –150°F. They also exhibit high chemical resistance to all chemicals except molten alkali metals and fluorine. As yet, they do not possess enough resilience to allow their applications as O-rings, so they are commonly designed in the shape of a chevron ring and are spread mechanically to provide the desired seal between the seal ring and the shaft or sleeve (Fig. 10).

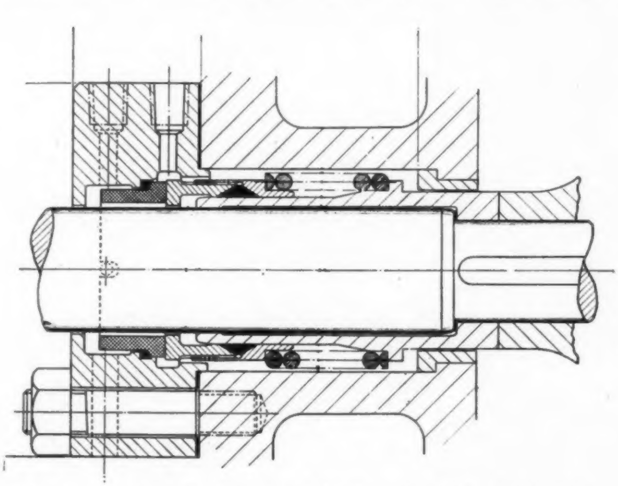


Fig. 10. Seal employing mechanically spread 'Teflon' wedge.

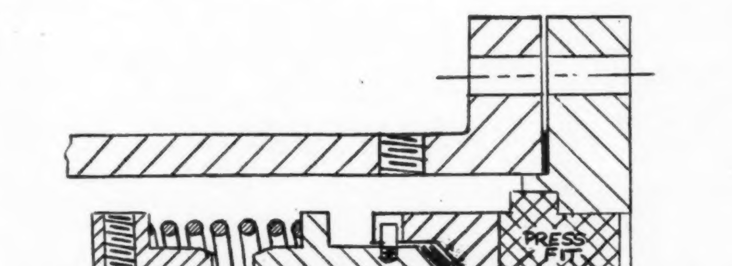


Fig. 11. Chevron-packing-type seal.

When operating at high temperatures, seals become more expensive and normally require a greater diameter because usually chevrons or tapered wedges are used which require a greater diameter to provide correct resilience and operation of seal.

Asbestos composition chevron rings are also used successfully as shaft seals in some types of seals (Fig. 11). These give good service, but their upper temperature limit is restricted to 350°F., since they contain synthetic rubber. They also deteriorate rapidly in the presence of acids, caustics and highly aromatic oils.

Square neoprene packings—cut to size on a diagonal joint similar to conventional soft packings—were extensively used before the development of the O-ring.

The rapid progress made in the synthetic materials field lends every hope that new materials will be developed which will extend the range of the existing types of mechanical seals up to much higher operating temperatures. At the same time it is not unlikely that seals which eliminate the critical resilient element will be developed or utilise designs which screen them from the full product temperature.

Spring

The spring behind the seal ring is usually designed to give a basic load to the seal ring, but some manufacturers use this spring to effect the drive. In this case the direction of the spring helices is very important. When this resilient spring drive is incorporated in the design of the seal, the seal ring moves continuously over the sleeve and prevents the O-ring from freezing in one position. Any inherent end-float of the pump shaft also assists in this continuous working.

Most fluids can be handled, although hydrochloric acid may lead to failure, for the choice of materials of construction for the spring is limited to those materials which are both suitable for spring manufacture and corrosion resistant.

In such cases it is practice to locate the spring on the outside of the pump supplying the necessary compressive load to the rotating seal ring. This is also standard practice in the food industry for hygienic reasons. A typical arrangement is shown in Fig. 12.

Bellows-type seal

A typical bellows-type mechanical seal is shown in Fig. 13. These are cheaper than the types illustrated in Figs. 2, 9 and 12, but are generally limited to the lower temperature, lower pressure applications. The material of construction of the bellows will restrict the upper operating temperature to about 200°F. and will be limited to those products which do not show chemical attack on the materials of construction of the seal and bellows.

The bellows are liable to be collapsed by high pressures, although modified bellows-type seals are available which overcome this difficulty. There are many applications for this type of seal which combine the advantages of economy, simplicity and easy unit replacement.

Effect of pump design

The design of the pump in which single seals are installed has a direct influence on the success of the seal operation and maintenance costs. A pump designed specifically for a mechanical seal has the impeller much closer to the pump bearing, thereby reducing the shaft deflection, due to the side forces developed by the hydraulic thrust on the impeller. Pumps of older design with a long shaft span between bearings accentuate the possibility of seal leakage caused by excessive shaft deflection. Cases have been reported with single volute cantilever shaft-type pumps where shaft sleeves have been severely scored by the throat bushing, although an original diametral clearance of .030 in. was given. This condition was observed only after the packing, which acts as a steady bearing, was removed and a mechanical seal installed.[2] In such cases it is standard practice to increase the shaft diameter to include the shaft sleeve, thereby increasing the rigidity of the shaft while at the same time eliminating the possibility of leakage along the pump shaft under the sleeve.

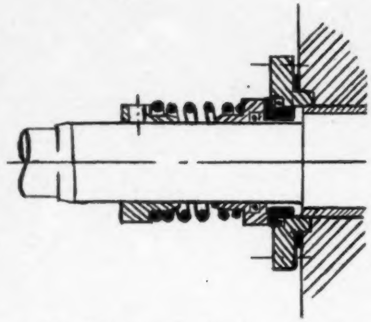


Fig. 12. Externally fitted seal.

Older process pumps with two stuffing boxes are gradually being replaced by the cantilever-shaft type with a built-in mechanical seal which substantially reduces the shaft overhang since the six to eight rings of packing plus lantern ring have been eliminated. Also the number of glands to be sealed is reduced to one.

Most pumps equipped with journal bearings have inherent shaft end float which was originally thought to be an adverse feature. Experience has shown that normal end float can be tolerated by most seals and is a desirable feature in that seal elements are continuously worked, thereby preventing them freezing into one position.

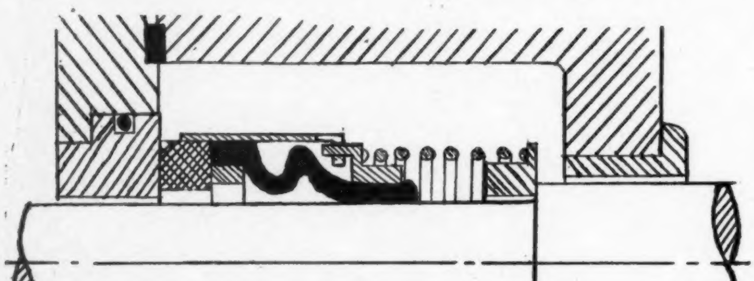


Fig. 13. Typical bellows-type seal.

When selecting a pump with a mechanical seal, it is important to check that the mechanical seal is easy to maintain. In this respect sufficient room must be allowed to work on the gland, and the end cover type of pump is particularly suitable in that the impeller may be removed with the minimum of work on the pump and adjacent piping.

A removal distance piece fitted between the motor and the pump will save much time and effort when maintaining the circumferentially split casing type of pump, allowing the pump bearing housing to be withdrawn with the minimum of disturbance in order to remove the impeller.

The author gratefully acknowledges the permission granted by 'Fluid Handling' and Dr. D. F. Denny to publish Fig. 1, together with the co-operation extended by Flexibox Ltd.

REFERENCES

[1]D. F. Denny, *Fluid Handling*, April 1953.

[2]J. A. Walmsley and H. A. Ward, Jun., *Petroleum Refiner*, Sept. 1953.

In the second part of this article, the author will discuss the applications of mechanical seals and the economics of installing them, and will give some practical advice on maintenance.

Petroleum Chemicals in Europe

The recent O.E.E.C. survey of Europe's chemical industry showed that, where petroleum chemicals are concerned, expansion is taking place as rapidly as is justified by likely demand and the economics of raw materials.

Total investment planned over the three years 1954-57 *amounts to* $196 *million, compared with a figure of about* $200 *million for* 1953-56. *Some of the investments planned in* 1953 *have since been realised,* e.g. *in the case of France and the Netherlands; on the other hand, some countries such as the United Kingdom have increased their investment plans. The largest proportionate increase planned in investment remains in Italy where present capacity representing an investment of* $25 *million should be increased in three years time to* $84 *million. A striking feature of the expansion will be the large-scale use of natural gas as a raw material* (355,000 *tons p.a. should be used when the plants planned are in operation). An interesting feature of expansion in general is the increasing use made of refinery gases.*

Boiler Feedwater Treatment and Oxygen Scavengers

By S. R. M. Ellis, PH.D., F.R.I.C., A.M.I.CHEM.E.
(Chemical Engineering Department, University of Birmingham)

Closer attention is being given to the treatment of boiler feedwater these days, especially with the higher pressure boilers such as are being used in power stations. In this article the author briefly surveys methods of removing impurities from feedwater, including ion-exchange processes, and then goes on to corrosion problems and the removal of dissolved gases. Oxygen is the chief ally of corrosion and the article finally deals with a subject that is of very great interest—the use of hydrazine as an oxygen scavenger in place of sodium sulphite.

THERE has been a steady increase in the working pressures of steam boilers of all classes in recent years. The higher operating pressures, which in some cases reach 2,000 p.s.i., have resulted in greater efficiency of power generation. Such developments have emphasised the need for high-purity boiler feedwater, since a low-solids and low-oxygen content in the feedwater decreases the amount of blowdown, avoids scale formation and reduces corrosion. Thus, in the case of high-pressure boilers, efficient water treatment is specified by the manufacturers of boiler equipment.

The chemical industry, however, is more interested in steam generation at pressures of 100 to 500 p.s.i. For such boilers the water treatment requirements are much less stringent. For pressures of 200 p.s.i. and more, when the total solids in the boiler is less than 500 p.p.m. and a large amount of make-up water is used, it is an advantage to have a high-purity feedwater.

The water purity problem

All natural waters are unsatisfactory for use in boilers, owing to impurities present and may be classified into hard and soft waters.

Hard waters contain 150 to 500 p.p.m. of dissolved solids and contain the bicarbonates and sulphates of calcium and magnesium. These salts, when precipitated during evaporation as magnesium hydroxide or as calcium and magnesium sulphates, form a scale on the boiler tubes.

Soft waters contain less than 100 p.p.m. of dissolved solids and give immediate lather with soap solutions. Hard and soft waters can contain 10 to 15 p.p.m. of carbon dioxide, 6 to 12 p.p.m. of dissolved oxygen, 0 to 50 p.p.m. of hydrogen sulphide and 0 to 40 p.p.m. of ammonia. Steam condensate water can contain 0 to 50 p.p.m. of carbon dioxide, oxygen 0.01 to 0.25 p.p.m. and ammonia 0 to 2 p.p.m.

High-pressure boilers operating at 650 to 2,000 p.s.i. require a feed concentration of less than 5 p.p.m. solids and an oxygen concentration of less than 0.005 p.p.m. Benson boilers in Germany operating at 2,000 p.s.i. have a feed with a solids concentration of less than 0.2 p.p.m.

Water treatment

The removal of calcium and magnesium salts to avoid scale formation can be achieved by:

(1) Lime and soda ash addition;
(2) The addition of phosphates;
(3) Evaporation;
(4) Zeolite-base exchange processes;
(5) Synthetic resin ion-exchange processes.

Lime and soda ash addition gives a softened water which can be further purified by evaporation or ion-exchange processes. Forbes[1] states that cold lime and soda ash treatment reduces both the Ca and Mg concentrations to 25 to 40 p.p.m., while hot lime and soda ash treatment gives 12 to 15 p.p.m. of Ca and 12 to 15 p.p.m. of Mg. After hot lime and soda ash treatment the total solids content of the feedwater may be 30 to 50 p.p.m.

Evaporation, in theory, gives a condensate free from solids. The disadvantage of evaporators is their relatively high capital cost and the fact that, in practice, foaming and entrainment results in the carry over of small amounts of mineral constituents with droplets.

With the development of high-capacity styrene-divinyl benzene co-polymers, and other synthetic ion-exchange resins, water of high purity can now be prepared by a combination of hot lime and ion-exchange operations.

Hiester and Phillips[2] claim that, for the production of high-purity boiler feedwater, the use of a combined hot lime ion-exchange operation gives lower operating costs than the older hot lime-and-soda phosphate process. De-alkalisation and demineralisation ion-exchange processes are also used.

Anion exchangers can remove chloride and sulphate ions and reduce the silica content of the water to <0.3 p.p.m. Silica can be a very troublesome contributor to scale formation in boilers and in cooling systems. At pressures above 550 p.s.i., silica can be carried over in the steam as gaseous silicic acid and can give rise to severe blade deposits in turbines.

Oxygen can be removed by stripping with steam in a de-aeration column, followed by chemical reaction with reducing agents or oxygen scavengers such as sodium sulphite or hydrazine.

Ion-exchange processes

Applebaum and McKeown[3] show that, when the solid content of the water is less than 100 p.p.m., the cost of demineralisation is less than that of evaporation. Thus ion-exchange processes are gaining in favour in the preparation of high-purity feedwater.

The use of high-purity feedwater should not necessarily be limited to high-pressure boiler practice. There is sometimes a case for the greater use of ion-exchange processes in the preparation of high-purity feedwater for low- and medium-pressure boilers. With a boiler operating at 200 p.s.i. and a boiler solids content of 400 p.p.m., the amount of blowdown can

be 5% or greater. If the feed is made up of 50% of condensate make-up water and 50% of softened water from the hot lime and soda treatment plant, the solids content of this boiler feedwater may well be 20 to 25 p.p.m. With this feed solid content figure giving a 5% or greater blowdown, there is an increased fuel bill due to the loss of heat in the blowdown water. The saving in fuel may well cover the depreciation and operating costs of an ion-exchange unit. The resulting high-purity water will also reduce the amount of scaling in the preheaters and economisers, etc. This statement does not, of course, apply generally, for it is dependent on the specific problem, the amount of treated water, and the total solid content of the feed and the boiler, etc.

Corrosion problems

Generally speaking, corrosion in pipelines, storage tanks, economisers and boilers can be caused by:

(*a*) The action of acidic groups present in the water or formed by the hydrolysis of salts present;

(*b*) Oxygen attack.

Oxygen is the prime factor in controlling the corrosion rate of ferrous metals in the average water system. Oxygen attack gives rise to a type of corrosion known as 'pitting,' which is attributed to the variation in the concentration of dissolved oxygen on the surface of the corroding metal. This attack, as the name implies, takes the form of a concentrated corrosive action upon small areas of the metal and can result in complete metal failure at the point of attack. The term 'oxygen attack' is slightly misleading in that it is not the areas of metal in contact with aerated water that are pitted but rather those portions which are starved of oxygen. The process may be briefly described as one of cathodic control of hydrogen ion discharge at an aerated metal surface, such that at any other point of the surface in electrical connection with the first, but not so freely oxygenerated, the electrons of (1) are provided by the dissolution of the metal as in (2), *i.e.*

$$2H^+ + 2e + \tfrac{1}{2}O_2 \longrightarrow H_2O \quad .. (1)$$

$$Fe \longrightarrow 2e + Fe^{++} \quad .... (2)$$

Thus the reduction of the oxygen concentration to a minimum and the prevention of oxygen concentration gradients at the metal wall can stop reactions (1) and (2). Furthermore, a protective oxide coating can minimise reaction (2). From a practical point of view, this localised pitting action is much more serious to the life of the metal than corrosion which spreads at a uniform rate.

In closed boiler systems, even though the oxygen concentration of the feed may be less than 0.15 p.p.m., this amount of oxygen significantly influences the corrosion rate, particularly since an increase in temperature accelerates the rate of attack. The importance of oxygen corrosion has been established not only by laboratory experiments but also by a considerable amount of practical experience and investigation.

Powell[4] has plotted the loss in weight of steel tubing with time for aerated and de-aerated water. With de-aerated water the rate of corrosion is slow, whereas with aerated water the rate of corrosion was much greater.

Straub and Ongman[5] have shown that the dissolved iron content of the water increased when free oxygen was present.

Fiss[6] reports that, in boilers operating at 900 and at 1,350 p.s.i., oxygen corrosion was serious and was arrested only by the complete removal of the dissolved oxygen.

Numerous instances have been quoted of corrosion of boiler tubes during shut-down periods. Often the tubes retain water when the boiler is drained and oxygen pitting takes place.

In 1949 a sub-committee of the British Iron and Steel Research Association made the following recommendations on the oxygen content permissible in boiler feedwater:

Boiler operating pressure (p.s.i.)	*Recommended oxygen content (p.p.m.)*
200 to 300	0.07 or less
300 to 500	0.03 or less
Above 500	0.015 followed by chemical scavenging

Forbes[1] considers that the oxygen concentration should be less than 0.007 p.p.m. in boilers and economisers.

Methods of removing dissolved gases

The three main methods employed to remove oxygen and dissolved gases are:

(1) Cold vacuum de-aeration.
(2) Hot de-aeration.
(3) Chemical de-aeration.

Condensers are responsible for de-aerating a large proportion of industrial boiler feedwater, particularly in closed systems used in power generation. Where steam is generated for use in a process, or where a 'pass-out' or 'back-pressure' turbine is used, additional make-up water must be added to the boiler and, if corrosion is to be prevented, the water must have the oxygen removed. Cold vacuum de-aeration reduces the oxygen concentration to 0.15 to 0.45 p.p.m., while hot de-aerators can reduce the oxygen concentration to 0.01 to 0.005 p.p.m. The carbon dioxide content is reduced to 0 to 10 p.p.m.

De-aeration equipment can be conveniently divided into three types:

(1) Tray de-aerator.
(2) Spray de-aerator.
(3) Impactor de-aerator.

In most of these designs of equipment the liquid phase is dispersed in the steam vapour phase. As the oxygen-water system is liquid-film-controlling, it is probable that the modern *Turbogrid* and *Kaskade* trays would give efficient vapour-liquid contacting. In comparing existing equipment the real difficulty is, however, that the existing literature is mainly descriptive with little or no published rate or efficiency data available.

The basic principle of all chemical methods is the introduction of a reducing agent to react with the oxygen. Since these agents are usually introduced after de-aeration, they are often referred to as oxygen scavengers. Typical reducing agents are alkali tannates, sodium sulphite and hydrazine.

Sodium sulphite as an oxygen scavanger

Although the patent rights on a process using sodium sulphite as a de-aerating agent were taken out in 1913, the method does not seem to have been exploited until several years after this date. The overall reaction is represented by the following general equation:

$$2Na_2SO_3 + O_2 \longrightarrow 2Na_2SO_4 \quad .. (3)$$

The data of Hitchens and Towne[7] and of Wickert[8] indicate those factors which influence the rate of reaction between sodium sulphite and oxygen. Oxygen is absorbed most rapidly by solutions of 4 to 10% in concentration, but the rate of oxidation decreases in dilute solutions. It is possible to accelerate the rate of reaction by adding minute amounts of catalysts. Some common reagents which show this influence are, in order of efficiency, the sulphates of copper, nickel, manganese, iron and cobalt. Indeed, it is suspected that catalysts in the form of impurities are always required to make the reaction proceed. If the sulphite is to be used cold, Forbes[1] claims it should be catalysed by the addition of about 5 p.p.m. of copper ion. A catalysed sodium sulphite, *Corrogen*, has been recently marketed in U.S.A.

The rate of reaction can also be influenced by the pH of the solution, excess sodium sulphite and temperature.

Although sodium sulphite effectively reacts with oxygen, recent developments have served to discredit the use of sodium sulphite. With the higher pressures and temperatures of modern boiler practice, Ongman[9] has shown that sulphite breaks down in the boiler to sulphur dioxide, thus giving rise to fresh corrosion problems. Zanchi[10] claims that the sulphite and sulphates can be deposited in the evaporator zone on the walls of the tubes and, together with the soda, form a mixture which melts and attacks the protective film of Fe_3O_4, thus exposing the iron to further attack. It is recommended that the concentration of sulphite in the boiler water should not exceed 5 p.p.m.

Shaffer[11] claims that, at high pressures with temperatures of 700°F. or higher, sodium sulphite is now being replaced by hydrazine. Sodium sulphite also has the disadvantage that it increases the solid or salt content of the boiler. This is a particular disadvantage for high-pressure boilers.

A modern problem, therefore, is to find a more satisfactory oxygen scavenger for the higher operating pressures of the future.

Hydrazine as an oxygen scavenger

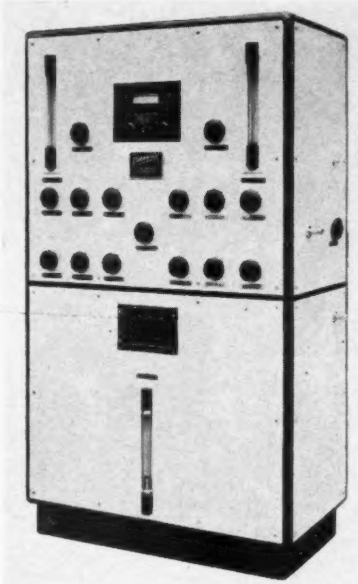
Hydrazine (N_2H_4) would appear to be one of the most promising oxygen scavengers, as it is a strong reducing agent capable of reacting with oxygen to give nitrogen and water as reaction products. Thus no solid products are formed and the solid content of the boiler water is not increased.

The reaction is:

$$N_2H_4 + O_2 \dashrightarrow 2H_2O + N_2 \quad .. \ (4)$$

In practice the reaction is not so simple, but this nevertheless describes the net result of the removal of oxygen. At saturation concentrations of oxygen in water at room temperature the reaction proceeds slowly. This is confirmed by the experimental investigations of Wickert,[8] Audrieth[12] and Moreland.[13] Thus, as in the case of sodium sulphite, the reaction with oxygen is not instantaneous at room temperatures.

Rate data at 20°C. have been published by Wickert,[8] while Moreland[13] has investigated various factors influencing the rate of reaction. The latter finds the rate of reaction is influenced by the pH of the water, the purity of the water, excess hydrazine, temperature and catalysts. Up to 70°C. the reaction rate increases to slightly more than double every 10°C. He finds that copper salts and activated carbon are the best catalysts. The reaction is catalysed at a surface. This is most important, since the reaction would proceed more rapidly at the surface of the metal pipe than in the bulk of the liquid. Thus protection against oxygen pitting is obtained and the effectiveness of hydrazine is not necessarily indicated by the oxygen content of the water.



[The Colborne Engineering Co. Ltd.

A feedwater demineralisation plant utilising columns of ion-exchange resins. The cation exchanger employed is a high-capacity styrene-base resin, and the anion exchanger a di-functional strongly basic quaternary resin. The latter material is specially adapted for removal of weak acids (silica and carbon dioxide). The combination of these exchangers, it is stated, will produce water possessing the high quality required, *e.g.*: Conductivity, below 1 m.mho; total dissolved solids, below 1 p.p.m.; carbon dioxide, practically nil; silica, 0.03 to 0.05 p.p.m.

The effect of hydrazine is to reduce the oxygen concentration at the surface of the metal to a minimum, to reduce oxygen concentration gradients and thus to minimise the previously discussed reaction (1) between hydrogen ions and oxygen. Hydrazine also appears to give a dense protective oxide film or scale which in all probability reduces the rate of solution of iron ions, *i.e.* reaction (2).

General use of hydrazine

In high-pressure boiler installations, because of the high temperature of the boiler feedwater, the hydrazine reacts rapidly with the oxygen. After thermal de-aeration, 50 to 300% excess hydrazine above the theoretical amount required is added to the feed and thus a small hydrazine residual maintained in the boiler. This residual hydrazine is vaporised or, if the temperature is above 350°F., partly vaporised and decomposed into ammonia and nitrogen. The decomposition occurs as follows:

$$3N_2H_4 = 4NH_3 + N_2 \quad ...... \ (5)$$

The presence of traces of hydrazine and ammonia in the condensate can be an advantage, since they help to maintain the pH between 8 and 9. Straub and Ongman[5] have shown that for a pH between 8 and 9 no iron or copper goes into solution. German, Italian and American industrial experience on boilers, economisers and condensers, operating up to 2,000 p.s.i., amply confirms these findings.

Hydrazine reduces the ferric oxide Fe_2O_3 to the magnetic oxide Fe_3O_4, which acts as a protective layer against corrosion. Hydrazine does not reduce the magnetic oxide Fe_3O_4 to ferrous oxide FeO.

Generally speaking, it is more convenient to effect mechanical de-aeration before the introduction of hydrazine, but there are special cases in industry where good protection against corrosion of pipelines and equipment is obtained by adding the hydrazine directly in the cold to the raw or purified water. An example of this is in the starting up of boilers and in the wet preservation of boilers during shut-down periods. Furthermore, hydrazine can also be used to reduce oxygen corrosion in hot-water-pipe systems operated with softened water. Often, too, in small low-pressure or medium-pressure boiler plants, it may not be feasible to install a mechanical de-aerator to reduce oxygen corrosion. The reason for this may be lack of space or the cost of equipment. When for such an application the feed temperature is less than 100°C. and the oxygen concentration is high, Bayer at Leverkusen recommend using the theoretical amount of hydrazine and passing the water through a carbon bed as catalyst. As previously mentioned, it has been found that activated carbon and other solid surfaces can greatly accelerate the rate of reaction.[13]

Zanchi[10] also recommends the use of a filter containing a special active carbon for catalysing the reaction. The carbon also assumes the function

of an auto-regulator, absorbing the excess hydrazine and restoring it when there is a deficiency. Furthermore, the filter extracts the copper in suspension in the water and thus a carbon filter is particularly recommended where the content of copper is high in the water.

Experience at the Tavazzano Power Station

One of the most interesting and valuable reports on the use of hydrazine at high pressures is that of Zanchi,[10] who describes the results obtained with hydrazine at the Tavazzano Power Station when using shell boilers generating steam at approximately 1,800 p.s.i. The steam temperature was 520°C. These results are of particular interest to power-station engineers in England, since a cylindrical shell-type (boiler) was used and copper was present in the circuit. It is proposed to summarise briefly some of the important practical points arising from this investigation at Tavazzano.

The feedwater after de-aeration contained 0.01 p.p.m. of oxygen, and approximately 400% excess hydrazine was added at a temperature of 130°C. The exact amount of hydrazine added could be varied to give a *p*H of 8.5 in the feedwater. Since oxygen is difficult to determine in the presence of hydrazine, a sample of feedwater was continuously run into a glass bottle in which was suspended a rod of polished soft iron. The water being analysed was maintained at 80 to 90°C. The efficiency of the hydrazine addition was confirmed if the surface of the iron remained bright. A residual of 5 p.p.m. of sodium sulphite was maintained in the boiler.

Using the photometric orthotolidine method of analysis, which detects oxygen to 0.001 p.p.m., no oxygen was detected in the saturated and superheated steam. Also there was no hydrazine in the steam and the ammonia content was 0.1 to 0.2 p.p.m.

Particular attention was paid to controlling the presence of copper in the circuit. In the boiler there was 0.02 p.p.m. and in the steam less than 0.002 p.p.m. of copper. The iron content in the circuit was 0.02 to 0.03 p.p.m. When the addition of hydrazine and ammonia was temporarily stopped the iron and copper content increased.

Thus it was concluded that the addition of hydrazine to the feed protects from oxygen the feed pump, the high-pressure preheater, the economisers, the boiler and the turbine.

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New Fatty Alcohols Factory in France

THE new French factory, inaugurated at Toulouse recently, for the manufacture of fatty alcohols by catalytic hydrogenation, is even larger than the plant established by Marchon Products Ltd. at their Whitehaven, Cumberland factory (CHEMICAL & PROCESS ENGINEERING, 1954, **35** (8), 228). It will be recalled that when the Marchon plant came into operation it was the largest unit in Europe for the manufacture of fatty alcohols.

The French have been making fatty alcohols since 1935, using the sodium reduction process. This is the standard process used in the United States, where metallic sodium is abundant and comparatively cheap. However, the French manufacturers, Société Sinnova, have been considering a change of method for some time, partly because of the high price of sodium and partly because the sodium reduction method can only be used for the glyceryl esters of the fatty acids and not for the acids themselves, their anhydrides or their metal salts. Sinnova have therefore been experimenting with a pilot plant using high-pressure hydrogenation (Marchon's new unit at Whitehaven uses a high-pressure hydrogenation process).

The cost of the plant is very high, but as cheaper raw materials can be used, hydrogenation has proved, in France as in Britain, to be more economical than sodium reduction.

The new factory has been built to produce by hydrogenation 7,200 tons p.a. of fatty alcohols (the new Marchon Products plant has a capacity of 4,000 tons p.a.). Sinnova count on exporting a big part of their production.

Toulouse, where the factory is situated, happens to be the town in which Prof. Sabatier, 55 years ago, discovered catalytic hydrogenation in his university laboratory. The site was chosen because it is near Saint-Marcet, where natural gas, a cheap source of hydrogen, is available. A detailed description of the new plant, which came into operation last October, was given by A. Bernard in *Chimie & Industrie*, November 1954.

It is intended to use pure hydrogen eventually, but in the meantime a mixture of 75% hydrogen and 25% nitrogen is being used for the synthesis of ammonia. Of the fatty raw materials the most important is coconut oil, transported from Marseilles by road or rail. The coconut oil, a white waxy solid, is warmed until it becomes liquid and then mixed by agitators with the catalyst, copper chromite, which is made in the factory itself. Both the gas and the mixture are then sent by high-pressure pumps through heat exchangers into the reactor, which is made of stainless steel. Here the esters are converted into fatty alcohols at a temperature of about 300°C. and a pressure of 4,300 to 5,000 p.s.i. Propylene glycol and iso-propyl alcohol are produced at the same time.

The reactor, which is 39-ft. high and has an internal diameter of 1 ft., is kept at the required temperature by electric induction heating. Gas and mixture enter at the bottom. The unused hydrogen—at present unfortunately mixed with nitrogen—is passed through the reactor again, after it has been separated from the liquid in a vertical column whilst still under pressure.

The liquid still contains the catalyst in suspension; it is withdrawn from the bottom of the column, degassed and filtered. The filtrate, a mixture of alcohols, is run into storage tanks and sent to another factory at Meaux-Beauval for the separation and purification of the constituents. (At this factory fatty alcohols are still produced by the sodium process.) The whole process is semi-automatic.

Britain's ATOMIC POWER Projects

★ **Twelve nuclear power plants are to be set up in the next 10 years at a cost of some £300 million.**

★ **New processing plants will also be needed eventually to deal with the increased quantities of fuel involved.**

★ **An interesting possibility is the increased use of thorium as a fuel instead of uranium.**

★ **Production of by-product plutonium might be an important factor in keeping down costs.**

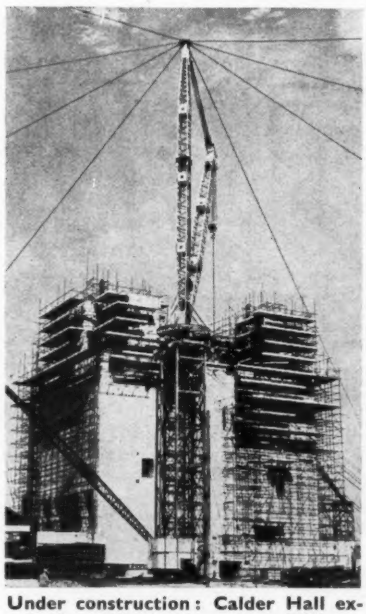
Chemical engineering skill of no mean order will be involved in the implementation of Britain's bold and enthusiastic plan for the production of atomic power in commercially useful quantities. The Government is going ahead with full-scale development not because all the technical problems have been solved, but in spite of them. Each individual unit in Britain's new atomic industry will present its own problems of design, construction and operation. Accordingly the British programme will be constantly modified in the light of new technical developments.

As well as the design and building of the reactors themselves and the development of materials to withstand the unprecedented conditions encountered, there are the problems involved in separating the fissile uranium, U. 235, from the fertile U. 238, in the chemical processing of the used fuel elements, and the extraction of plutonium from them.

The atomic factories at Windscale, Capenhurst and Springfields already bear witness to the pioneering work that has already been done by chemists and chemical engineers towards the creation of Britain's newest and most exciting industry.

'AS a leading industrial nation our duty, both to ourselves and to other countries, is to establish this new industry of nuclear energy on a firm foundation and to develop it with all speed. It is a major industrial development that will bring with it revolutionary changes in technique. We shall only learn the new techniques by pressing forward with the practical applications wherever we can and in spite of the many uncertainties surrounding each enterprise.'

It is in this spirit that Britain's Government has laid its plans for the development of atomic energy. The provisional programme, set out in a White Paper,* covers the next ten years in some detail and gives an indication of the probable developments in the following ten years. It will be constantly modified as time goes on and at each stage final decisions will not be taken until the last possible moment so that new technical developments can be used to the fullest advantage.



Under construction: Calder Hall experimental atomic power station.

Practical objectives

Future developments are likely to be directed at two main objectives: using the main nuclear material, uranium, more efficiently; and reducing the capital cost per kilowatt of a nuclear power station, in terms both of the construction of the reactor and of its initial charge.

During the next ten years two types of reactors are likely to be brought into use on a commercial scale. The first type will be similar to those now being constructed at Calder Hall, but improvements in design during the period should enable the later models to show a great advance in efficiency compared with the earlier ones. They will be gas-cooled, graphite-moderated 'thermal' reactors using as fuel natural uranium or slightly enriched uranium. The first improved models could be designed and built so as to come into operation in about six years' time.

The second type of reactor that may be built for commercial operation during the next ten years is a liquid-cooled 'thermal' reactor. This type requires more complicated techniques which at present would result in higher costs. But with further developments liquid-cooled reactors should be able to give a much higher heat rating than the first gas-cooled reactors for the same capital cost. They might therefore prove more economic than the gas-cooled reactors, although the comparison will depend on how much the gas-cooled type can be improved. They could take any of several forms, most of which need enriched fuel, and could use for this purpose the plutonium produced in the earlier reactors in conjunction with natural uranium. The first commercial liquid-cooled reactors might be built during the latter part of the next ten years and begin operating about 1965.

Development after 1965 may take several forms: thorium may be used, at first in conjunction with plutonium, as an alternative fuel; 'homogeneous' and 'fast breeder' reactors may be developed. (It has already been decided to build a full-scale model of a 'fast breeder' reactor capable of producing power on a site at Dunreay in Caithness.)

Twelve power projects

The provisional programme for the construction of nuclear power stations† is as follows:

The construction of two gas-cooled, graphite-moderated stations (each with two reactors) would be started about mid-1957. These stations should come into operation in 1960-61.

The construction of two further stations would be started in 1958-59. These would also have two reactors

*'A Programme of Nuclear Power,' (Cmd. No. 9389), H.M.S.O., 9d.

† The term 'station' is used here to denote the smallest unit that is likely to be built. In practice more than one such station may be built on the same site.

each and would be similar in type to the earlier two stations, but should show an improved performance, particularly in heat rating. Each of the eight reactors in these early stations would have a net output of electricity of 50 to 100 megawatts, so that the total output from the four stations, which should all be in operation by 1963, would be somewhere between 400 and 800 megawatts.

The construction of four more stations might perhaps start in 1960, and then a further four 18 months later, say, 1961-62. These might come into operation in 1963-64 and in 1965. The White Paper states that it is difficult to specify what type of station these would be, but it is probable that each station would consist of only one reactor, which would be much more highly rated than the reactors in the first four stations. The stations begun in 1960 might be developments of the gas-cooled, graphite-moderated type. The last four stations might be of the liquid-cooled type which might by then have been developed sufficiently to be economically satisfactory. The total installed capacity of the eight stations in this group should be well over 1,000 megawatts.

On the assumption that nuclear stations would be used as base-load stations, they would by 1965 be producing electricity at a rate equivalent to that produced by about 5 to 6 million tons p.a. of coal.

New processing facilities

The present ancillary plant, which has been built and is used primarily for military purposes, will be adequate at first for a commercial programme of this magnitude, but some expansion will be necessary later. A new fuel processing and fabricating plant will be needed in due course in addition to the existing factory at Springfields to meet the rapidly increasing demand for nuclear fuel, and a new chemical processing plant will eventually be needed to deal with the large quantities of used fuel taken out of the nuclear power stations. Such slight enrichment as may be necessary for the fuel elements in the early stations can be provided from the existing capacity of the diffusion plant at Capenhurst.

Long-term prospect

The possibilities of expansion will depend to a great extent on the speed with which the necessary techniques are mastered by industry at large. One possible limitation on the rate of expansion in the later years is the supply of nuclear fuel, particularly the more highly enriched material that will be needed for some of the advanced types of reactor. By the late 1960s the early reactors should be producing plutonium in quantity and this would be available for the later reactors. The provisional programme and the further expansion thereafter will also call for increased supplies of uranium, and no doubt other countries will be increasing their commercial demands at the same time.

Recent evidence suggests that uranium is more plentiful than was once thought; considerable workable deposits of medium-grade ore are known, while the widespread existence of low-grade ores implies that adequate quantities can be produced from them if necessary.

Moreover, the expansion in the requirements of uranium should be mitigated by the greater economy in its use that will by then have been achieved and by the possible development of the substitute fuel, thorium, which should be available in considerable quantities if it is required.

Plutonium and costs

Developments in reactor design, such as the introduction of liquid cooling, should gradually lead to much higher heat ratings without much increase in capital cost.

The fuel cost depends on three things: (1) the cost of the raw material, uranium; (2) the processing cost, including the conversion of ore into fabricated fuel elements, the chemical processing of the used fuel elements and the extraction of plutonium from them; and (3) the level of irradiation, *i.e.* the amount of heat that can be got from each ton of fuel in the reactor before it has to be taken out.

It is expected that it will be possible to extract as much as 3,000 megawatt-days of heat from every ton of fuel. This is the equivalent of the heat from 10,000 tons of coal. There is as yet no practical experience of this level of irradiation at high temperatures and the metallurgical behaviour of the fuel elements is uncertain. But there are many lines of development which should overcome such metallurgical defects as may appear.

On the question of costs, the White Paper points out that some credit should be allowed for the fissile by-product plutonium. It is in many ways equivalent to uranium 235, another form of fissile material. But plutonium can be extracted by chemical means from a power reactor's used fuel for only a fraction of the cost of separating uranium 235 from natural uranium in a diffusion plant. When concentrated fissile material is available in quantity there will be great scope for the design and development of more advanced and more efficient reactors that need enriched material and will not operate on natural uranium. For example, most types of liquid-cooled reactor need enriched material and, looking further ahead, concentrated fissile material in the form of either uranium 235 or plutonium is required for a 'fast breeder' reactor or for starting a thorium system. In this manner the early reactors will be producing not only electricity but also the capital equipment (*i.e.* the initial charge of fissile material) for future power stations. Without the plutonium it would not be possible to build up a system of nuclear power stations of steadily advancing efficiency.

In the early stages of an expanding nuclear power programme it is to be expected that concentrated fissile material will be scarce and that, if there were a free market, its price would be high. It will be required for enriching the fuel charge in new commercial reactors, and also for many experimental and development purposes.

Heavy Water Enterprise in New Zealand

Some days after the White Paper on Britain's atomic power projects was published it was announced that the United Kingdom Atomic Energy Authority and the New Zealand Government had formed a joint enterprise to produce heavy water and electric power from geothermal steam bores at Wakeiri in the North Island of New Zealand. The board of directors includes Mr. A. S. White, head of the Chemical Engineering Division of the Atomic Energy Research Establishment, Harwell.

Production of heavy water by late 1957 is envisaged.

It was previously announced that the designing of the dual purpose plant would be undertaken in the United Kingdom (CHEMICAL & PROCESS ENGINEERING, 1954, **35** (9), 295), and that the first unit to be installed at Wakeiri will produce 40,000 kw. of electric power.

The new development would be important to Britain's atomic power plans at a later date if heavy water reactors were used to any extent. It was the need for a high-efficiency low-cost packing in the distillation process to be used at Wakeiri which led to the development of *Spraypak* expanded metal packing (see page 91).

How Drops are Formed from Gas or Vapour Bubbles

NEW LIGHT ON LIQUID ENTRAINMENT

Liquid entrainment is encountered in various unit operations and its effects are in many cases detrimental. So far there has been little information as to the size and size-distribution of entrained drops or on the mechanisms by which they are formed by ebullition or other processes. At a meeting of the Institution of Chemical Engineers in London recently, three workers presented the results of a study of drop formation due to bubbles of gas or vapour travelling upwards through a liquid and collapsing at the liquid-gas interface. The sequence of events associated with the collapse of the bubble has been traced and the sizes, trajectories and number of drops formed have been determined. Some observations on the function of baffles in entrainment separators were recorded and the conditions giving rise to re-entrainment defined. The authors of this paper were Prof. D. M. Newitt, M.C., PH.D., D.SC., F.R.S. *(past president of the Institution), N. Dombrowski,* M.ENG., PH.D., G.I.CHEM.E., *and F. Knelman,* M.ENG., PH.D. *Here is an abridged version of the paper.*

A BUBBLE of moderate size (say 0.5 cm. diam.) rising through a liquid usually assumes an ellipsoidal form and rocks in a vertical plane about the longer axis. The terminal velocity of ascent is, over a wide range of sizes, nearly independent of size. When the bubble reaches the surface of the liquid it usually rebounds back and forth with decreasing amplitude until, just before collapse, it comes to rest with its upper part projecting above the surface in the form of a hemispherical dome. The walls of the dome are extremely thin at the apex, but thicken towards the base.

The collapse of the bubble is associated with a release of energy sufficient to impart a comparatively high velocity to any drops formed and to give rise to a wave of disturbance in the surrounding liquid.

It is important to know by what mechanism the bubble breaks up and the origin of the drops formed. Various conflicting views have been expressed. Strong evidence has been brought forward recently that a thin liquid film breaks up by the initial formation of a number of perforations which subsequently expand to give a lace-like structure. The liquid ligaments so formed are unstable and break into small drops of varying size.

Mechanism of bubble collapse

From studies carried out with high-speed photography, the mechanism of collapse appears to be as follows:

The bubble coming to rest at the liquid surface forms the hemispherical dome, its internal pressure producing a depression of the interface. Liquid drains from the dome until the upper part is so weakened that the internal pressure causes the formation of a secondary cap. This cap subsequently disintegrates, giving rise to droplets of a few microns in diameter. These droplets, which are too small to be seen in the photographs, are carried away by the rush of gas issuing from the perforated dome. The result of these events is to set up a system of standing waves, and to leave a well-defined crater in the interface. As the crater fills in, the momentum of the inflowing liquid produces a jet, which rises at high velocity and in certain circumstances detaches one or more comparatively large drops from its apex. It is these drops, which may have a diameter of the order of 0.1 cm., that are responsible for the main losses by entrainment. The jet then retracts and the surface of the liquid returns to rest.

Experiments were carried out with air bubbles from a glass capillary at various depths below a water-air surface. The results showed that the diameter of the large drops produced decreases nearly linearly with decreasing bubble diameter. The number of these drops increases with decreasing bubble diameter.

Both the diameter and the number of large drops decrease with rise in temperature.

The very small drops produced by the initial disintegration of the bubble do not travel vertically upwards but are scattered by the explosion. It was not found possible to take accurate measurements of diameter and number of drops formed, although the diameter decreased with increasing bubble diameter whilst the number increases.

The rising jet has been the subject of experiments which give results in close agreement with theoretical calculations for the height to which the jet rises, the velocity of the rising jet, and the distance and time of break-up of the jet.

Surface tension seems to be the main property in determining the number of large particles, the number being proportional to the square root of the surface tension. The diameter of the large particles is probably proportional to some inverse power of the bubble diameter and, since smaller bubbles produce faster jets, this inverse proportion to a power of the bubble diameter is not unlikely.

The function of baffles in entrainment separators

In many types of entrainment separators a system of baffles is employed to deflect the gas stream so that entrained drops are carried by their momentum on to the baffle whilst the gas passes round it. The drops striking the face of the baffle form a liquid film which drains to the lower edge of the baffle. The film then reforms large drops which fall out of the gas stream and may be collected.

Several possible sources of re-entrainment present themselves in the above situation. Firstly, the drop may be atomised by impact on the baffle face and the resultant smaller drops re-entrained. Secondly, drops may be atomised by the gas stream during their period of formation and detachment at the baffle edge. Lastly, the drops may be re-entrained as they fall from the baffle edge. At sufficiently high gas velocities large drops may become unstable and atomise.

In effect, a baffle acts as an agglomerator—producing large drops from smaller ones which have impinged on its face. A knowledge of the size of

drops detaching from baffle edges is of interest, since several investigations have established the critical size for instability of drops in gas streams. It is also important to know the size of detached drop as a function of the thickness and shape of baffle edge and of the velocity of the gas stream.

To obtain data on the behaviour of baffles a wind tunnel was constructed. It consisted of a rectangular perspex duct fitted with a constant-head liquid feed device and adjustable baffles. A calibrated Venturi throat was attached to the duct, enabling air velocities to be measured.

The baffles were constructed of brass plate of different thicknesses, each baffle having one square edge, one 90° vee-shaped edge and one 45° bevelled edge.

The arrangements were such that drops of predetermined size could be injected into the gas stream and, when fully accelerated, allowed to impinge on the baffle; or liquid could be supplied at a constant rate to one face of the baffle and allowed to drip off its lower edge.

Photographic methods were used for recording the events associated with the operation.

Individual drops of various sizes were projected into the air stream and allowed to impinge on the baffle face. The air velocities were raised from 610 to 2,130 cm./sec. and a Kodak high-speed cine-camera was used to record the results. In addition a large number of visual observations were made of drop impact. In no instance was there any evidence of rebound or break-up; the drop on impact deformed to a flat disc which subsequently spread to a thin film.

Measurements of drop sizes from the baffles showed that the diameters at first decreased with increase in baffle thickness (for all types of edge), passed through a minimum at about 0.22 cm. plate thickness, and then increased. For the square-edged baffle the increase was fairly considerable up to a plate thickness of about 1.2 cm., after which it tended to a constant drop size of 0.8 cm. diameter. For the vee-edged baffles there was only a slight increase in diameter with thickness from the minimum of 0.4 to 0.47 cm. at plate thickness of 1 cm. and upwards.

Re-entrainment from baffles

By high-speed photography the mechanism of re-entrainment was studied in the wind tunnel. Below velocities of about 1,200 cm./sec. the air stream does not materially affect the mode of detachment of the drop. At greater speeds the air stream tends to tear the drop from the drip point, producing an elongated jet which detaches, and may or may not produce a satellite. The process is similar to atomisation from spinning discs. At velocities above 1,800 cm./sec., atomisation increases and several satellites are formed. At the highest velocity, 2,340 cm./sec., atomisation is continuous, drops being torn off from the main drop as fast as liquid pours into the drip point. Moreover, at this velocity, drops tend to be unstable and may be stretched to a thin film or bag with a thick rim. The rupture of this film will then produce the same systems of small and large drops as do bursting gas bubbles.

Conclusions

It is possible to control to some extent the incidence of entrainment. By a proper control of bubble diameter it is possible to operate under optimum conditions. This may be illustrated by comparing the two bubble diameters 0.53 and 0.311 cm. in an air-water system. If 1 cc. of vapour is considered, there will be 13 bubbles of the large diameter producing a total of 2.33 large drops having a mean diameter of 0.1 cm. and 156 small drops having a mean diameter of 22 microns. The same volume will produce 64 of the smaller bubbles, generating a total of 63 large drops of mean diameter 0.074 cm. and 100 small drops of mean diameter 58 microns.

In general, the elimination of large drops is fairly simple. In evaporation it may be accomplished by increasing the vapour space and in plate columns by increasing the plate spacing.

Bubbles generated at shallow depths (0.64 cm.) produce fewer large drops. The depth at which a bubble is generated affects the trajectory of the main drop it forms on bursting. By choosing depths at which the velocity of the resultant drop has a strong horizontal component, vapour spacing could be reduced and baffles arranged near the interface could be employed with advantage. It has also been shown that small drops have a flat trajectory on a high initial velocity and here again some form of splash deflectory should be advantageous.

Increase in temperature produces a significant reduction in the number of drops produced in an air-water system. A reduction in surface tension also reduces the number of drops.

The minimum baffle thickness which should be used is 1.9 cm., from which water drops will be detached with a diameter of 0.78 cm. Square-edged baffles should be used whenever possible, since the drop size is considerably greater from this type of edge.

A consideration of the physical dimensions of the system and the ballistics of the drops should enable the optimum number, size and arrangement of the baffles to be determined.

New Uses for Industrial Waste Materials

IN all research on waste utilisation there are certain common factors which come into consideration. These are:

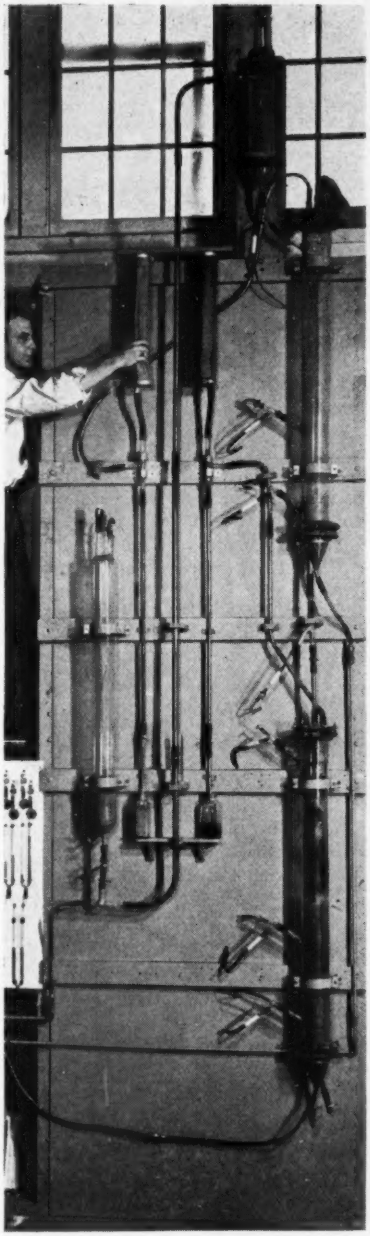
(1) Physical and chemical characteristics and volume of waste.
(2) Properties of material to be studied for utility.
(3) Laboratory evaluation of material for which potential uses were seen.
(4) Field work with industry, including consumer tests.

During these phases—some conducted simultaneously—the economic feasibility of the product must be constantly examined.

The American non-profit-making Stanford Research Institute has been working for several years toward the productive use of waste materials ranging from radioactive fission by-products to the purification of sewage water for irrigation. Some of these projects are briefly noted below.

Chemicals from sawdust. One recent waste-utilisation project was the development of an inexpensive method of converting wood wastes into useful materials. A process was devised in which the waste was held in fluidised suspension while subjected to heat. Under optimum conditions the process produced attractive yields of chemicals, carbon and gas.

Pelleting redwood. Another project, undertaken for the Hammond Lumber Co., involved finding a profitable use for redwood bark. Many potential uses for the fibrous bark were surveyed and the production of charcoal appeared promising.



FLUIDISATION DEMONSTRATION UNIT

A laboratory unit that is being used by chemical engineers of the Stanford Research Institute, U.S.A., for developing a new process whereby carbon and a range of chemical by-products are produced from the fluidisation of sawdust and other wood wastes. The unit has two purposes: (1) to demonstrate the principles of the fluidised carbonisation process and (2) to determine optimum particle-size distribution of the solids, velocities of the gas stream and dimension of fluid beds. On the left can be seen part of the control board for regulating the flow of fluids and solids. The cylinder at left centre on the framework is the feed hopper. It contains raw sawdust. The other two glass cylinders, upper and lower right, contain fluid beds. The twin cylinders at the top left (handled by technician) are bag filters, which collect solids, and immediately below them are small glass cyclones, which separate gases from solids.

A study of current charcoal production methods showed certain deficiencies. The research effort then concentrated on developing a process to overcome these problems. Laboratory experimentation demonstrated that the simultaneous application of heat and pressure could mould light redwood bark into a relatively dense pellet. By carbonisation a firm briquette could be produced. As no commercially available pelletising machine could be modified to produce the redwood pellets, chemical engineers designed and constructed a pilot machine to briquette the bark.

Utilisation of wood residues. Another project, undertaken for Pope & Talbot Inc., was concerned with the utilisation of wood residues near Oakridge in southern Oregon. Because of a high incidence of conk rot (*Fomus pini*) in these stands, it was estimated that the area had an excess of 30 million ft. of slash residue and 10 million ft. of mill residue per year. This wood, defective for the better grades of lumber, makes some form of residue utilisation necessary to realise fuller use of the forest resources and to help bear the additional cost imposed by the necessity of clearing the faulty timber from the land.

Various existing and proposed methods and processes for wood-residue utilisation were reviewed by a team of economists and chemists of the Institute and discussed with experts in the field. Each process was considered with respect to its application at Oakridge. These considerations included geographical location, water supply, transportation, raw material supply and marketability of product.

From the survey of products and processes for utilisation of residues, the research team concluded that feasible products for Oakridge residues are: semi-chemical pulp (converted to corrugating material), hardboard, chipboard, wax (extracted from bark) and soil conditioners. The study further indicated that the most profitable overall utilisation may require the integration of several processes, each supplementing the others. Some types of residue are potentially more valuable than others. Clean chips are worth more as a source of hardboard or pulp than as fuel or soil conditioners. On the other hand, sawdust, shavings and bark are difficult to utilise in a pulp mill, but will serve well as fuel or for conversion to ligneous soil conditioners.

Coke from low-grade coals. For the coal industry of western United States a study has been made of the utilisation of low-grade coals to produce a char (coke) which would be a satisfactory substitute for high-temperature industrial coke. This survey of known processes strongly indicates that, by adequate modification of carbonisation methods, a low-temperature process could be developed to produce a coke equivalent to, or better than, that produced by high-temperature processes. It is known that some low-temperature processes are able to carbonise coal at lower cost than high-temperature methods and yield greater quantities of valuable tar by-products.

Sewage purification. Considerable thought and study has been given to the purification of sewage for irrigation purposes by the use of continuous ion-exchange methods as developed by chemical engineers. Principal emphasis was put on the feasibility of reclaiming sewage water from San Francisco and the Peninsula area and using the water for irrigating in the Santa Clara Valley of California. It was determined that for technical and health reasons sewage water must undergo two treatments before demineralisation, leading to irrigation. These treatments are: a primary treatment to settle grit and organic solids, plus skimming to remove grease and scum; and an oxidation treatment to produce a stable effluent having little or no undissolved solids.

A survey of the area determined that only one major sewage source—the Richmond-Sunset plant—would meet irrigation water standards after secondary treatment. The other major sources give an effluent which is of poor mineral quality, making it unacceptable for general irrigation use.

Dust from steel furnaces. Work for the steel industry involved the installation of dust collectors on open-hearth furnaces. This resulted in the capture of a small daily tonnage of very fine material for which uses were found in paint pigments, rubber compounding and in smelting operations.

PVC gloves and clothing. Seven new illustrated leaflets from James North & Sons Ltd., numbered 100 to 106 inclusive, feature PVC gloves, industrial foul-weather PVC clothing, North PVC headgear, etc.

New Chemical Plants for Britain

THIS and the coming months will see the bringing into operation of many new British plants for the manufacture of chemicals and kindred products. The chemical industry's productivity has been raised by about 40% since 1948, chiefly through very heavy capital expenditure. A recent Financial Times *report suggests that something over £450 million has been invested since the war in the chemical and allied industries, excluding oil refineries, and the current rate of investment is some £75 million p.a. The new plants coming into operation this year, together with the steady increase in the efficiency of existing plant, will lead to an increase of perhaps 6 to 7% in productivity.*

Imperial Chemical Industries alone had spent £210 million up to the end of 1953 on its capital programme, with capital commitments of £60 million still outstanding. The largest single I.C.I. project scheduled for completion in 1955 is the £20-million Terylene *plant at Wilton. The beginning of production in this unit was reported in* CHEMICAL & PROCESS ENGINEERING *last month, and some further details are given in this issue. Other I.C.I. facilities due to be brought into operation or completed this year include plants for making Perspex, polythene, nylon polymer, silicones, titanium sponge and argon. Much interest centres on the Nobel Division's new 4,000-ton-p.a. pentaerythritol plant, to come into operation in the spring. A new formaldehyde plant is scheduled to start production at about the same time. The second oil cracker at Wilton and the plant for making butadiene products are also making good progress.*

Monsanto Chemicals' major projects are the introduction towards the end of the year of plants at Ruabon for the manufacture of maleic and phthalic anhydride and further extensions to the oil additive plant at Newport. There seems to be a possibility that the 4,000-ton-p.a. synthetic rubber plant at Ruabon will also come into operation before the year is out.

The biggest developments within the Distillers' group in 1955 are to be in the field of plastics materials on the 54-acre site which British Geon and British Resin Products share at Barry, Glamorgan.

These are only a few of the new developments, and many other prominent concerns will be bringing in plants for manufacture of products ranging from sulphuric acid to insecticides.

On this and the following pages we publish details of new plants, which have come into prominence during the past month or so, for producing greases and polyvinyl acetate, as well as the new Terylene *plant already referred to.*

POLYVINYL ACETATE

IN a new plant set up at a cost of some £500,000 at Chester-le-Street, Durham, the British Oxygen Co. is undertaking the preparation of stabilised vinyl acetate and the manufacture of polyvinyl acetates in granular form. Both these products have previously been imported. Starting from acetylene and acetic acid, the stabilised monomer is produced and is then converted to polyvinyl acetate (PVA), one of the most versatile materials used in the plastics industry.

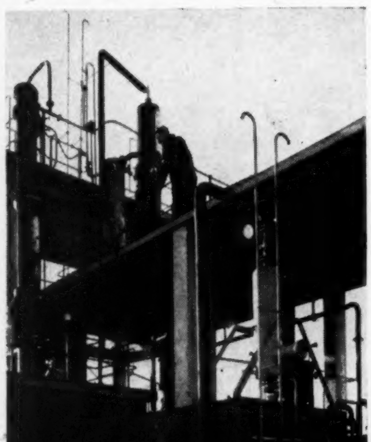
Already makers of carbide cyanamide and acetylene, British Oxygen decided at the end of the war to use these products in the synthesis of other chemicals. The first venture was the production of melamine derived from cyanamide, which itself comes from carbide. Now PVA is being manufactured to take advantage of the large production of acetylene, while vinyl pyrrolidone is being made by a special technique of which the company claim to be the only exponents in the United Kingdom.

Never before has PVA been manufactured in the U.K. from home-produced vinyl acetate monomer, but now production of vinyl acetate and its conversion to the various types of PVA can be controlled in one plant from raw materials to finished product.

Three types of product are available: stabilised monomer, emulsion polymer and granular polymer.

Production of the monomer

Vinyl acetate is prepared by interaction of acetylene (also prepared at Chester-le-Street and transferred to the PVA plant through an $\frac{1}{8}$-mile-long pipeline), and an excess of vaporised acetic acid under the influence of temperature and pressure in the presence of a catalyst:



Top of the monomer distillation section of the new polyvinyl acetate plant.

$$CH_3COOH + CH \equiv CH \rightarrow CH_3COOCH = CH_2$$

The resultant product is distilled primarily to recover the surplus acetic acid, but three main fractions are obtained containing, respectively, vinyl acetate, compounds of lower boiling point and those of higher boiling point.

The plant for the manufacture and purification of the monomer takes up a large part of the site, and the top of the distillation section can be seen above the top floor over 80 ft. above ground level.

In view of the potential dangers from acetylene and vinyl acetate fumes, this part of the building is exposed to the atmosphere and is thus always subjected to a stream of fresh air. With the plant exposed to such an extent the desirability of keeping the numbers working on it to a minimum is obvious. In effect the entire unit is fully automatic. All separation, distillation, cooling and pumping is done mechanically and is supervised by remote control from a central electronic instrument room. This is slightly pressurised to prevent any collection of inflammable gases being fired by the sparking of electric terminals. The same applies to all electrical wiring, which is sealed and kept at a pressure slightly above atmospheric to prevent entry of explosive vapours into the circuit and their ignition by shorting.

The purified vinyl acetate is a volatile unstable liquid. When required for sale in this form, a copper derivative or hydroquinone is added as a stabiliser.

Polymer production

In a separate section the vinyl acetate is polymerised. Unlike the monomer plant, this section is enclosed and air-conditioned to avoid contamination from dust, etc., and to maintain a control of temperature.

The unit runs continuously, with the staff working three 8-hr. shifts. Apart from temporary stoppages to replace catalysts, cleaning and maintenance, the plant has been running continuously for several months.

To make the granules, the pure monomer is mixed with a catalyst and other chemicals. This mixture is stirred in tanks until the reaction is complete, when the viscous water-white odourless polymer is collected and extruded through $\frac{3}{16}$-in. die heads. The polymer is solidified on a cooling conveyor and cut into granules $\frac{1}{8}$-in. long.

A suction pump draws them away and whirls them to the top of an inverted cone to throw out dust and lighter particles, allowing the heavier ones to fall through a revolving four-blade valve into a hopper from which they are bagged for sale.

The granules are a useful form of PVA, because they are 100% polymer, are convenient for storage and transport and are dissolved in a variety of solvents depending upon the particular use for the material.

For the emulsion the pure monomer is mixed with a catalyst emulsifying and thickening agents, etc., to produce the appropriate emulsion, collected in tanks and kept at a controlled temperature ready to be barrelled for sale.

Applications of PVA

Polyvinyl acetate has a wide range of uses in the paints, adhesives, paper and textile industries. At the moment, most of the sales are of emulsion products, but great interest is being taken in PVA granules by printing ink manufacturers and the most recent developments have occurred in the adhesive and polish industries.

Plastics raw materials and the intermediates from which they are made are sectors of the chemical industry which are growing fast in Britain, Western Europe and America. The trend will undoubtedly continue and the use of PVA will increase with it. At Chester-le-Street there is room on the site for additional plant if required.

Heart of the new grease manufacturing plant at Birkenhead is the first floor, seen here, where the four contactor heaters and eight kettles are situated.

GREASES

By 1939, the Birkenhead works of the Vacuum Oil Co. had been established as one of the largest oil and grease production units in Europe. Two years later, nearly all its buildings and manufacturing equipment were destroyed or damaged by enemy action. Rebuilding was immediately started and temporary plant erected. Total reconstruction was, however, completed only in 1953.

The reconstructed plant incorporates the most modern methods and equipment, and it is now once again one of the finest and largest in Europe. Bulk oils are brought in by deep water and coastal tankers, which discharge their cargoes through pipelines into storage tanks near the waterfront. These stock oils are processed and despatched as finished products by rail and road transport.

During 1954, 1,500 to 2,000 tons/week of finished products were despatched.

Grease plant

When the original grease plant was destroyed in 1941, a temporary one was set up on a different site. By September 1941, this makeshift plant, originally designed to manufacture 80 to 90 tons a week, was soon producing about 200 tons of grease a week under the most difficult conditions. It continued to do so until the new plant, which was started in 1951, was finally completed in 1953.

The new grease plant manufactures no less than 200 different grades. A survey of grease manufacturing plants in Europe and the United States was carried out, and from this the basic design of the new plant was decided. It was designed to satisfy the foreseen market requirements and also to allow for a 50% increase in capacity.

In general terms, greases are soap-thickened lubricating oils. They vary in consistency from mobile fluids to solids harder than ordinary household soap. To manufacture grease, first a soap must be made and then dispersed in lubricating oil. Soap is made by boiling together fat or fatty acid with an alkali such as lime or caustic soda or some other metallic base. In the new grease plant, saponification takes place in a contactor vessel, final grease manufacture in a kettle.

Packaged raw stocks, *i.e.* fats and alkalis, are received and stored in the ground-floor warehouse. From here these packaged stocks are transferred, as required, by lift truck and elevator to the top floor, where they are prepared before being fed to the manufacturing vessels. Liquid stocks such as mineral oils, used in the manufacture of practically every grease, and the fats used for soap bases are stored in the tank farm adjacent to the plant. From this tank farm the oils and fats are pumped through pipelines to the manufacturing floor. The infra-red heater is used for fats which require

melting into the steam-heated dump tanks. The liquid fats are then passed to weigh tanks before reaching the manufacturing kettles.

Details of vessels, etc.

The nerve centre of the entire plant is the first floor where the contactors and kettles are situated. The raw materials are charged to a contactor, which is heated to about 550°F. by the hot oil circulating system, and saponification begins. Pressure is generated in the contactor and, when the process is completed (in about 20 min.), the contents are discharged to one of the two adjacent kettles which it serves. Other components and the necessary amount of mineral oil are stirred into the soap concentration in the kettle until the required standard is achieved; this final stage varies according to the product from 1 to 5 hr.

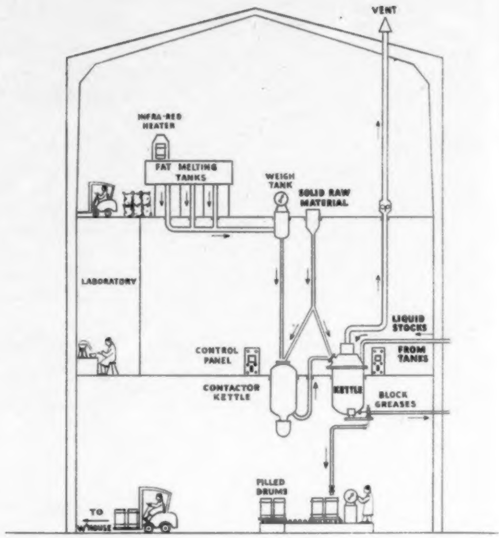
All control instruments are on this floor as well as the laboratory, pilot plant and offices.

The chief manufacturing vessels are the four Stratco contactor heaters (three 6,000-lb. and one 3,000-lb.), and the eight kettles (four 15,000-lb., three 8,000-lb. and one 2,000-lb.) which they serve. The size of the kettles and contactors used is determined by manufacturing demands. The contactor vessel, manufactured by the Stratford Engineering Corporation, U.S.A., is of original design. It provides for the rapid circulation and dispersion of the contents. It has a high-speed impeller driven through the bottom of the vessel which moves the contents over three heating surfaces. This ensures very high heating rates. The contactor is heated by S.V. heat transfer oil, which is circulated through an oil-fired tubular heater at a temperature of about 600°F.

Four of the kettles (two 15,000-lb. and two 8,000-lb.) are heated by steam and four by oil. The internal surfaces of the kettles are machined and the contents are mixed by contra-rotating paddles fitted with scrapers to assist heat exchange and prevent local overheating. All the vessels have instruments which ensure that correct temperature and process time are maintained during manufacture, and all liquids from the storage tank farm are controlled by automatic shut-off meters pre-set to the required volume. The manufacturing vessels also have ventilating systems on top of them which prevent any fumes or odours from escaping during manufacture.

The pilot plant, also supplied by Stratford Engineering, is a miniature of the main manufacturing unit: it has a 100-lb.-capacity contactor and a 400-lb. kettle—and is used for development operations.

Simple flow diagram of grease plant.



The vertical oil heater used for heating the fats fed to the contactors, which has already been mentioned, has a capacity of over 10 million B.Th.U./hr., and the system takes about 4,500 gal. It is automatically controlled and incorporates the latest protective devices. It, too, is fully instrumented, and there are duplicate relays on the grease manufacturing floor so that the operators there can tell at what temperature and pressure the heater unit is working.

Laboratory approval, packaging, services

The laboratory occupies part of the office block on the manufacturing floor. When approval has been given to the batch of grease, the outlet valve at the base of the kettle on the ground floor is opened and the finished grease is pumped through filters into pre-stencilled packages. A Cornell grease homogeniser on the ground floor is used to reinforce the mixing in the kettle for some grades of grease. The filled packages are removed by fork lift truck to a cooling area where they are finally sealed and taken to the warehouse or the loading bay.

The control of essential services such as steam, air, water and liquid stocks and supply pumps is fully covered by a central control console supplied by New Weston Ltd. containing instruments manufactured by the Bristol Instrument Co., and installed on the first floor.

Transfer and filling-off pumps are of the rotary, positive-displacement type, and were supplied by Messrs. Stothert & Pitt Ltd. Where necessary, these pumps are steam jacketed. The hot oil circulating pumps used in conjunction with the oil heater are centrifugal type and water cooled. They were supplied by Hayward Tyler & Co.

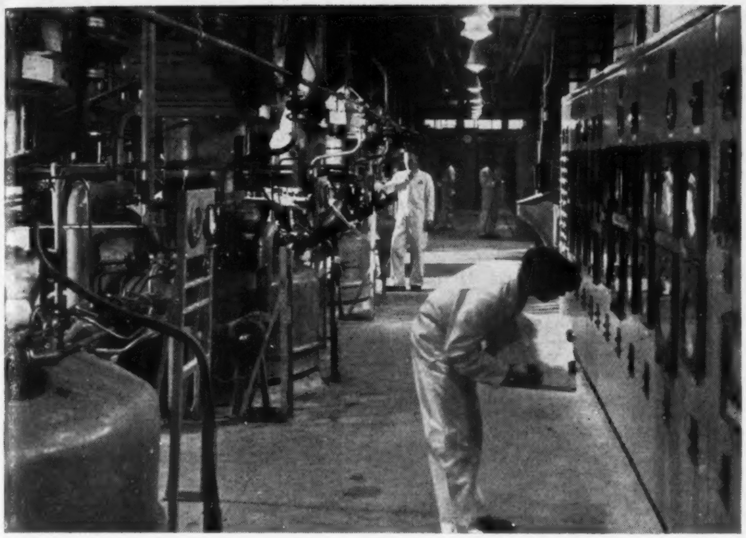
Other suppliers of pumps were Pulsometer Engineering Co. Ltd. and Sigmund Pumps Ltd., while Broom & Wade Ltd. supplied air compressors. Manufacturing kettles were supplied by W. J. Fraser & Co. Ltd. and the oil heater by Head Wrightson Processes Ltd. Head Wrightson were the main contractors.

'TERYLENE'

LAST but not least in this trio of new plants is Imperial Chemical Industries' big *Terylene* factory at Wilton in the North Riding of Yorkshire, which we described briefly in February. The main raw materials are para-xylene, ethylene glycol and methanol. All these are products of the I.C.I. Billingham Division, and the first two are made at Wilton works.

In the first chemical building of the *Terylene* plant, para-xylene, which is made from a petroleum distillate, is converted to terphthalic acid—a white, crystalline substance.

In the second building, the terphthalic acid is processed with methanol, forming dimethyl terephthalate, which is then polymerised. Polymerisation takes place in autoclaves at high vacuum and at high temperatures. The result is polyethylene terephthalate (*Terylene* polymer).



The autoclaves in which 'Terylene' polymer is made.

The polymer is extruded in the form of a ribbon from the autoclave on to a 'casting wheel.' This ribbon of polymer solidifies on the casting wheel and is then cut into chips for easy handling, and conveyed by suction to the spinning building. Here the polymer chips are dried to remove residual moisture, and are then put into hopper reservoirs ready for melting and conversion to either filament yarn or staple fibre.

It was towards the end of 1943 that I.C.I. was approached by the Calico Printers' Association, in whose laboratories *Terylene* was discovered, and asked to help in developing the fibre. In 1947, I.C.I. acquired the world rights to manufacture *Terylene* (except in the United States, where the rights had already been acquired by E. I. du Pont de Nemours & Co.). Experimental plants were followed by a pioneer polymer plant and a pioneer spinning plant, the aim of which was to produce sufficient quantities of the fibre for thorough commercial appraisal. Until recently, the production of *Terylene* has been confined to the output of these pilot plants, which are currently producing at the rate of approximately 2 million lb. p.a. The capacity of the first unit of the Wilton plant will be 11 million lb. p.a., divided equally between filament yarn and staple fibre. When the second unit of the plant comes into operation the capacity will be doubled.

I.C.I., through Canadian Industries (1954) Ltd., is building a plant at Millhaven, near Kingston, Ontario, to manufacture *Terylene*. Annual capacity will be 11 million lb. and the capital cost will be more than 20 million Canadian dollars. More recently, I.C.I. has concluded negotiations with a number of European firms, whereby *Terylene* will be manufactured under licence in Italy, France, Western German and the Netherlands.

Tests on 'Spraypak' Tower Packing

Last year it was reported that a new industrial distillation- and absorption-tower packing, Spraypak, *had been developed at the Atomic Energy Research Establishment, Harwell. The new packing was described and illustrated in* Chemical & Process Engineering *of July* 1954. *It consists of a cellular structure made for most purposes from commercial* $\frac{1}{8}$*-in. nominal mesh,* 20 *to* 24 *s.w.g. expanded metal of* $\frac{1}{16}$*-in. strand width. Tests were carried out using air-water rigs and an M.S. distillation column.*

Some further facts about Spraypak *are contained in a paper presented to the Institution of Chemical Engineers, in London, by J. A. McWilliams, H. R. C. Pratt, F. R. Dell and D. A. Jones. Some of these are as follow.*

For convenience, aluminium mesh has been used for most of the work, but it is envisaged that in most instances stainless steel would be used in commercial plant.

In air-water tests at low liquid rates and gas rates approaching zero the liquid runs as a more or less continuous film over the cell walls, while at higher liquid rates some of the liquid tends to stream through the packing mesh from cell to cell. As the gas rate is increased the streaming ceases and the liquid film is disrupted with the formation of bubbles.

Above about 20% of the flooding rate the film begins to be detached from the upper portion of the cell walls and spraying sets in. The trajectories of the spray droplets become inclined more and more as the gas rate is further increased, and at angles somewhat above the horizontal a limited amount of entrainment from lower to higher courses occurs.

In distillation tests at total reflux, where the mass flows of liquid and vapour are the same, the liquid flows on the strands of the mesh, around the mesh holes, at the lowest boil-up rates. At 20 to 25% of the flood point, the strands overload and partial filming and bubbling starts. At slightly higher rates spray formation sets in, and is virtually complete over all the surfaces at about 35% of the flooding point.

The density of the spray and the contacting efficiency increase steadily up to the flood point.

In air-water tests an extra course above the feed point acted as an effective de-entrainer.

The effect of variation in cell height and pitch on the flooding rate was also examined. For cells with approximately 45° wall angles, the flooding rate varies as the 0.44 power of the course height within the range 2 to 6 in.

Reducing the wall angle, *i.e.* increasing the pitch for the same course height, slightly increases the pressure drop and very slightly reduces the flooding rate, but improves the liquid distribution.

Typical distillation data were obtained and plotted in the form of H.E.T.P. and pressure drop per theoretical plate against percentage flooding rate. It appears probable that the limiting values of H.E.T.P. at the flood point are substantially independent of pitch, and inversely proportional to course height, corresponding to a constant course efficiency in the latter case. The shape of the H.E.T.P. curve depends to some extent upon the packing geometry, however, probably as a result of variations in liquid distribution. Thus the liquid film appears to increase in thickness as the pitch is increased, resulting in flatter H.E.T.P. curves. Reducing the course height at the same time has the effect of reducing the H.E.T.P., although at the expense of a somewhat reduced throughput.

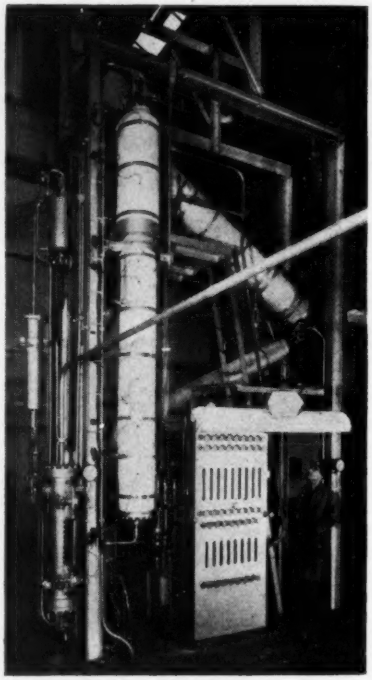
Fluidised Pilot Plant for the Fischer-Tropsch Synthesis

Work on the Fischer-Tropsch synthesis at the Fuel Research Station, D.S.I.R., led to erection of a pilot plant for the study of various process techniques. Experience gained with this plant, using a fluidised catalyst system, has paved the way for extensive pilot-plant work using the 'slurry' system. At a recent meeting of the Institute of Petroleum, the fluidised pilot plant was described by C. C. Hall, M.SC., PH.D., F.R.I.C., *and A. H. Taylor,* M.B.E., B.SC., M.I.MECH.E. *The results obtained in two runs of 14 and 30 days' duration were presented and discussed. Below is an abstract of the paper.*

THE development of improved processes for the synthesis of liquid fuels and chemical products from carbon monoxide and hydrogen necessitates research along two general lines. One involves the study of catalyst activity and selectivity and the effect of the process variables (temperature, pressure, gas composition, etc.) on the rate and course of the reaction. This type of work can, in general, be carried out in simple, small-scale tubular reactors containing a bed of catalyst granules or pellets. The other line is concerned with the development of a suitable reaction system for carrying out the exothermic and highly temperature-sensitive reaction on a commercial scale. Although a valuable preliminary assessment of the suitability of a particular system can be obtained in equipment with a reaction space of, say, 1 to 2 litres, it is ultimately essential to build and operate a pilot plant of sufficient size to provide a more reliable indication of the probable performance obtainable in a full-scale plant.

In the work on the Fischer-Tropsch synthesis at the Fuel Research Station, two reaction systems, which appear to have advantages over the original fixed-bed system, have been studied. These are the fluidised catalyst system and the liquid-phase (' slurry ') system.

In 1949 it was decided to erect a pilot plant for the study of various process techniques on a scale of 1 to 2 barrels of products per day. At that time, the fluid-catalyst technique appeared to be particularly promising, and operation of small units had provided sufficient information to permit the design of a larger unit. It was decided, therefore, to build the pilot plant for operation, in the first instance, with fluidised catalyst. The potential advantages of the slurry system were fully recognised, but further work on a smaller scale appeared desirable before constructing a pilot-scale reactor for this process.



A general view of the reactor showing the differential pressure panel.

The general plan was the installation of the necessary equipment for purification, measurement and compression of 2,000 cu.ft./hr. of synthesis gas and for the handling of a product output up to 80 gal./day. A change in the particular synthesis technique employed would, therefore, require only a change in the reaction vessel and certain items of equipment directly associated with it—a comparatively minor proportion of the whole plant in terms of cost and labour.

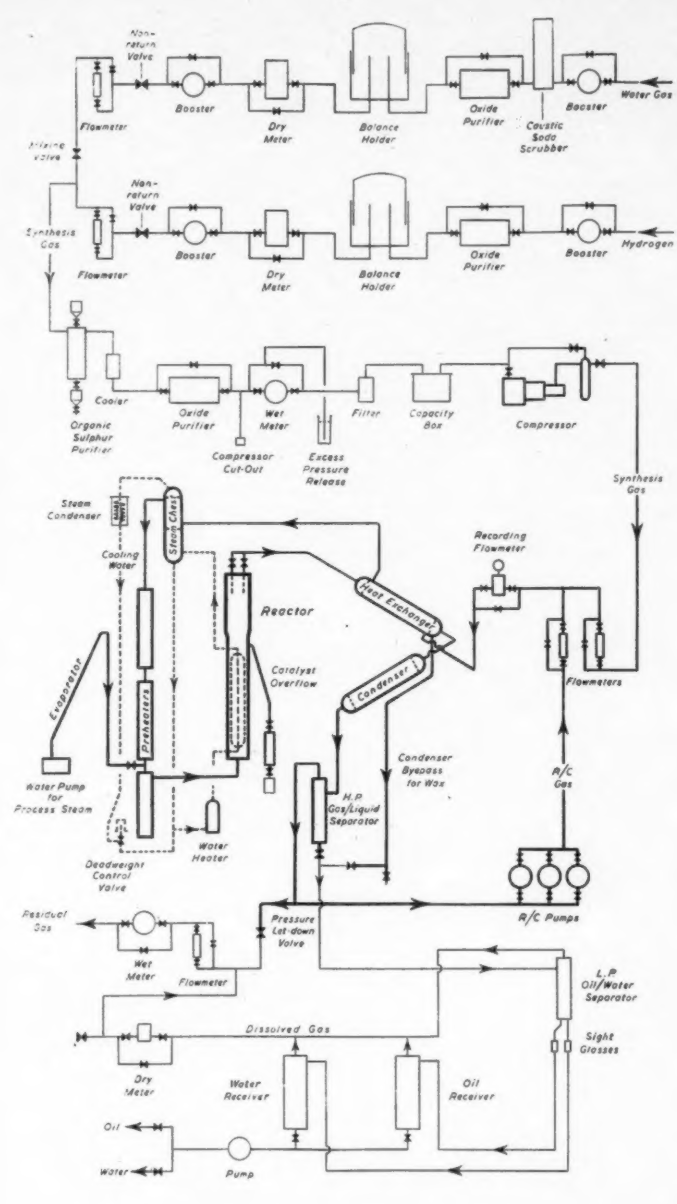
By the time the plant was ready for operation (September 1952), it had become apparent that the dense-phase fluidised-catalyst technique possessed certain limitations and disadvantages and was probably not the most suitable one to employ where coal is the basic raw material. It appeared, furthermore, that the fluid-catalyst synthesis plant built for Carthage Hydrocol Inc. at Brownsville, Texas, had run into serious difficulties, certain of which, apparently, were associated with the synthesis reactors. This could be interpreted as evidence that pilot plants of a similar size to that built at Greenwich had proved to be an unreliable basis for the design of full-scale plants. As, however, no details of the design or performance of either the pilot plant or the full-scale plant built in the U.S.A. have been published, it was considered to be desirable to obtain some first-hand knowledge of the fluid-catalyst synthesis process when operated on a pilot-plant scale.

Two test runs

The plant was designed for a throughput of fresh feed gas up to 2,500 cu.ft./hr. (s.t.p.), operating pressures in the range 20 to 50 atm. and reaction temperatures up to 340°C. Parts of the plant coming into contact with condensing products were made in 18/8/1 stainless steel; all other parts in mild steel. The feed gas was obtained by blending blue water-gas and hydrogen from the steam-iron process. Both gases were available on the site in adequate quantities; they were purified to remove hydrogen sulphide and were led by pipeline to the plant building. A simplified flowsheet for the entire system is shown in the diagram.

An alkali-impregnated mill-scale catalyst of the type used in previous experiments in smaller reactors was employed. For the assessment of performance, the runs were divided into test periods each of approximately 24 hours' duration.

Data representative of the peak performance achieved during Test Run 1 show that, of the total product obtained during the selected period, methane and C_2 hydrocarbons, which are not



Pilot Scale Fluidised Catalyst Hydrocarbon Synthesis Plant

Flow Diagram

regarded as recoverable products, amounted to over 25% and that, of the recoverable products (C_2 and higher), nearly 20% consisted of water-soluble chemicals. Propylene and butylenes, which together amounted to over 20% of the total product, are, clearly, major products of this type of operation.

A rapid collapse in catalyst activity occurred during the last phase of Run 2. The precise cause of the final collapse has not yet been established. The sulphur content of the catalyst increased after Period 8 of the run at a much greater rate than would have been predicted from the sulphur content of the feed gas and, at the end of the run, was more than twice the calculated figure. It is considered unlikely, however, that the uptake of sulphur to the extent indicated by the catalyst analysis would have caused such a complete loss of activity.

It is possible that the final collapse in activity was due to cloaking of the catalyst surface by a layer of very stable, complex carbonaceous material which prevented the access of either hydrogen or synthesis gas to the surface.

Comparison with results obtained in a small reactor

The gas conversions obtained in both pilot-plant runs were lower than had been anticipated from previous experience with a 1-in.-diam. reactor, and it was only in Run 2 that the product distribution was of the expected type. It was of interest, therefore, to carry out experiments in the 1-in.-diam. unit with samples of catalyst withdrawn from the pilot-plant reactor. After Run 1, experiments were carried out with a sample of reduced catalyst taken from the reactor before starting synthesis and with an average sample of the catalyst discharged at the end of the run. The results were compared with those of a run with the fresh catalyst which formed part of the charge for Test Run 1. (A run in the empty 1-in. reactor at a pressure of 30 atm. and a temperature of 330°C. showed no volume contraction or change in the gas composition.) It was not always possible to reduce the recycle ratio in the small unit to as low a value as that obtaining in the pilot plant. Hence comparison of catalyst activity in the two units must be based mainly on the CO conversion, which is not sensitive to changes in recycle ratio. The results show that a particular sample of catalyst exhibits a definitely higher activity in the small unit than in the pilot plant, the difference being particularly marked with the catalyst discharged at the end of the pilot-plant run. The results also suggest that the product distribution obtained in Test Run 1 was a function of the mixed catalyst employed and not one of plant scale or geometry. The product distribution obtained with the fresh mill-scale catalyst in the 1-in. unit was broadly similar to that obtained with fresh catalyst in the second test run in the pilot plant.

In two runs in the 1-in.-diam. unit with freshly reduced mill-scale catalyst operated under conditions similar to those used in Test Run 1, no change in CO or ($CO + H_2$) conversion was obtained in a period of 15 days.

Data showing the activity in the small unit of samples of catalyst withdrawn from the pilot plant at various stages of Run 2 were also obtained. The freshly reduced catalyst and that

taken from the pilot plant after Period 22 show a significantly higher activity in the 1-in. unit. The activity of catalyst taken after Period 8 is, however, little different from that observed in the pilot plant during Period 8. The extremely low activity in the small unit of the catalyst recovered after the final shut-down is qualitatively in agreement with experience in the pilot plant, as is also the complete failure of re-reduction in hydrogen to effect any improvement in activity.

In the light of all the data obtained it must be concluded that under a particular set of operating conditions a batch of catalyst will give a significantly better performance in the 1-in.-diam. unit than in the 8-in.-diam. pilot plant. In view of the fact that the height-diameter ratio of the fluid bed is of the order 25 to 30 in the 1-in. unit and 10 in the pilot plant, this conclusion would have been predicted from investigations by other workers on gas mixing in beds of fluidised solids. This work showed that, owing to the 'escape' of reactants in the form of bubbles which pass rapidly through the bed, the extent of conversion of gaseous reactants will be less than in a fixed bed, and will decrease as the height-diameter ratio of the fluid bed decreases.

Other workers also studied the oxidation of NO to N_2O_4 in a fluidised-bed system and found that, with H – D = 10.4, the conversion obtained was 95% of that obtained with H – D = 27, and with H – D = 6 it was only 85 to 89%. The effect of height-diameter ratio became more marked with increase in the level of conversion.

Conclusions

Although the runs were marred by mechanical troubles, they established the soundness of the main design features of the plant and achieved the desired objectives. Periods of up to 350 hours' continuous operation with high gas conversions and product yields and with a low rate of carbon formation were achieved. The product distribution was apparently fairly typical of that obtained in American 'Hydrocol' pilot plants. Much more work would be required in order to obtain reliable data for catalyst life. The cause of the complete collapse in catalyst activity in Run 2, after 30 days on stream, has not yet been clearly established. If this represents the maximum life which can be expected of the type of catalyst used, when operated with pure gas and without interruptions and changes in conditions, the catalyst consumption would amount to 1 lb. (unreduced catalyst) per 72 lb. (about 10 gal.) recoverable products. It is considered, however, that the mill-scale catalyst has a much greater 'productivity' than this figure suggests.

The observed change in the oxygen content of the products with time is clearly a function of catalyst ageing in a 'batch' operation and, if operated with continuous catalyst replacement (or regeneration), the catalyst in the reactor would reach a steady-state condition, and products of constant quality should be produced.

The experiments provided the type of thermal and other data required for the design of a full-scale plant, but the comparison of the synthesis performance obtained in the pilot plant with that obtained in the small unit, taken in conjunction with published work on the characteristics of fluid-solids systems, suggests that it would be unwise to design a full-scale reactor by a simple increase in dimensions of the pilot-plant reactor. It appears that, if a full-scale Fischer-Tropsch reactor using a dense-phase fluid catalyst is to reproduce the performance obtained in a pilot-scale reactor, the height-diameter ratio of the bed must remain unchanged. This could be achieved by using, on the full scale, a multiplicity of relatively narrow tubes (*e.g.* 2 to 4 in. diam.), containing the fluid catalyst, within a single pressure-resisting shell. Such an arrangement has been patented in America, but without any reference to the point discussed above.

The experience gained in the operation of this pilot plant will be of value in planning and carrying out similar work with a reactor designed for the slurry process. This process, which is considered to be of greater promise for the Fischer-Tropsch synthesis than the dense-phase fluidised process, will be the subject of more extensive pilot-plant work than that described in this paper.

RECENT PUBLICATIONS

Clarifier. The K 21234S clarifier marketed by Alfa-Laval Co. Ltd. is described and illustrated in an 8-page brochure. The clarifier has a capacity of 3,350 gal./hr. and has a multi-cylinder bowl with special large sludge space of 14½ gal. (170 lb.). Solids are removed from the bowl in a very dry condition and the machine is fitted with a centrifugal paring device which delivers clarifier spirit under pressure, so obviating the need for a separate pump. It is of robust construction and is complete with clean and dirty spirit glasses, speed indicator, pressure gauge, etc.

Knife edges. In answer to the problem of wear and tear of knife edges on analytical balances, Stanton Instruments Ltd. have produced a 'synchro-release' mechanism which automatically controls high-speed release and arrestment. This device, it is stated, eliminates the possibility of impact damage on knife edges. Full details are given in an illustrated 4-page folder.

Monel and Nimonic alloys. A new series of data sheets is being issued by Henry Wiggin & Co., presenting, in concise form, essential physical and mechanical data on wrought high-nickel alloys. The second in the series, dealing with Monel, a corrosion-resisting nickel-copper alloy of wide engineering application, has just been issued. The first in the series dealt with Nimonic 90 for springs A further data sheet issued by the company contains supplementary data on Nimonic 90 and Nimonic 95

Electronic process control. A recent publication (No. 272) of Evershed & Vignoles Ltd. contains a 12-pp. survey, illustrated with photographs and diagrams, of various types of panels and control desks which have been supplied by the company recently. It features installations for crude distillation units, SO_2 extraction plant and re-run units, and catalytic polymerisation plants, and explains the principles of operation of the Evershed process control system. Another illustrated publication, No. 295, gives comprehensive details of the Evershed process controller Mark 3.

Antioxidant for rubber. In many applications a white or coloured rubber is required to show the minimum of discoloration on exposure to light. A new antioxidant, *Nonox* WSL, has been introduced by the Dyestuffs Division of I.C.I. and is described as powerful and completely non-staining. A 12-page illustrated brochure detailing the tests made on *Nonox* WSL and their results, and containing physical and full technical data, has been issued by the Dyestuffs Division.

Separation of Volatile Materials by Gas-Liquid Chromatography

By A. T. James, PH.D.
(National Institute for Medical Research, Mill Hill, London)

Gas-liquid chromatography is a rapid, highly efficient and relatively simple means of separating volatile materials and should be applicable to all substances capable of being distilled. At present it is a versatile analytical tool, but there seems no reason why it should not be scaled up to deal with larger quantities. Dr. James is co-inventor of the technique with Dr. A. J. P. Martin, F.R.S., *a pioneer of chromatography and Nobel prizewinner. Here the new technique is described and its possibilities discussed.*

CHROMATOGRAPHIC methods for the analysis of mixtures are used in almost every research laboratory and chemical works in the world because of the high resolving power of the method. The greatest triumphs of the technique have been obtained with both water-soluble and fat substances of biological origin, in particular amino acids, vitamins and sugars. Before the introduction of liquid-liquid partition chromatography by Martin and Synge, many attempts were made to separate gases and vapours by chromatographic methods using a stationary solid adsorbent and a moving gas. The frequent occurrence of non-linear distribution isotherms, however, prevented clear-cut separations and the technique never came into general use.

In their first paper on the liquid-liquid chromatogram in 1941, Martin and Synge suggested that separations of volatile materials should be possible by the use of columns having a stationary liquid phase and a moving gas phase. This suggestion was, however, never taken up until 1951, when James and Martin[1] developed the technique for the separation of the volatile fatty acids. As the mobile phase of the chromatogram is a gas, it is possible to use long, thin columns and still obtain high rates of flow in distinction from the liquid-liquid chromatogram. Furthermore, the gaseous nature of the moving phase makes diffusion and hence attainment of equilibrium the more rapid and, since rate of diffusion is the main factor controlling the efficiency of chromatograms, gas-liquid columns of very high efficiencies can be produced. In addition the columns are normally used at temperatures between 65 and 200°, these elevated temperatures making the rate of attainment of equilibrium even more rapid. It is clear, therefore, that high efficiencies (columns 4-ft. long having efficiencies between 1,000 and 2,000 theoretical plates) can be obtained even at high rates of flow of the mobile phase, so that refined separations can be carried out often at high speed. The technique possesses the further advantage that it is often easier to detect small concentrations of vapours in gases than it is to detect small concentrations of substances in solution.

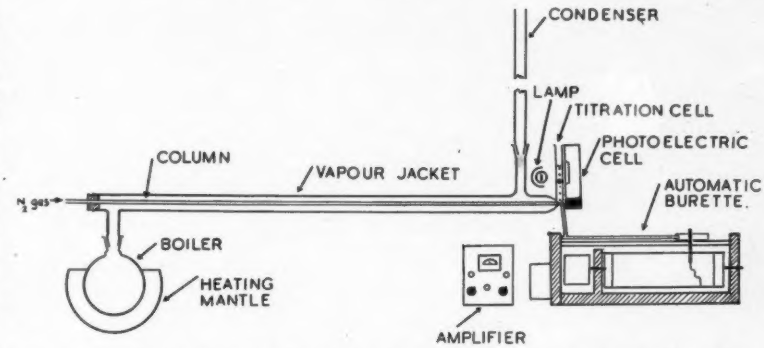


Fig. 1. Schematic layout of gas-liquid chromatogram using automatic titration as the means of detecting substances leaving the column.

Experimental arrangement

The experimental arrangement used is to distribute the appropriate stationary phase (of low vapour pressure at the temperature of operation of the column to prevent its loss) over the surface of a finely divided solid (usually kieselguhr, diatomaceous earth), this being packed into a glass or metal column maintained at the required temperature by a vapour jacket. The mixture to be separated is applied to the top of the column packing and is then blown down the column by a stream of permanent gas, usually nitrogen, from a source of constant pressure. The substances pass down the column, move apart according to their relative volatility in the stationary phase chosen and, provided the column is long enough, emerge from it in the nitrogen stream separate from one another, when they are detected by a suitable device. The columns may be used repeatedly without any obvious signs of deterioration.

For substances that ionise in solution, *e.g.* fatty acids, aliphatic and aromatic amines, pyridine bases, etc., titration provides a simple means of detection and quantitative estimation. In Fig. 1 is shown a schematic diagram of the apparatus used by James and Martin.[1] The column is held in a horizontal vapour jacket to maintain its temperature constant (water being used in the boiler for a temperature of 100°C. and ethylcellosolve for a temperature of 137°C.) and the gas on leaving the column passes into a titration cell containing a suitable solvent, usually water, and a *p*H-sensitive indicator (phenol red). As soon as an acid (or base) emerges from the column it is trapped in the water and the resultant change in *p*H causes the indicator colour to change from red to yellow. A green-sensitive photo cell situated behind the titration cell res-

ponds to the change in colour by a change in photo current; this is passed to a simple amplifier and causes the drive to a recording burette to start and so deliver dilute alkali (or acid) to the titration cell until the indicator colour returns to red, when the burette drive is switched off. A ball pen attached to the moving plunger of the burette records its movement on a chart attached to a drum rotating at known speed. In this way a graph is obtained showing a series of steps; each step denotes the appearance of an acid (or base) and the height of the step gives directly the amount of material (Fig. 2). A horizontal line on the graph denotes no titratable material emerging from the column. After loading the columns, turning on the nitrogen stream and filling the titration cell with indicator solution the whole process is automatic.

Separations achieved

In Fig. 2 is shown the separation of acetic, propionic, *iso*- and *n*-butyric acids completed in 30 min., using a column 4-ft. long, of internal diameter 4 mm., with a stationary phase of MS 550 silicone oil containing 10% w/w of stearic acid. The upper curve is obtained by graphical differentiation of the experimental curve and shows the zones of acid in a form more familiar to users of chromatographic techniques. In Fig. 3 is shown the separation of acetic, propionic, *iso*-butyric acid, *n*-butyric acid and all the isomers of valeric acid, the separation taking only 100 min. In this case a column 11-ft. long was used, being folded into a hairpin shape so that it could be accommodated in the same vapour jacket as the 4-ft. column. The efficiency of this column is 2,000 theoretical plates, a figure undreamed of by distillation chemists. The sensitivity of the method is such that amounts of acetic acid as low as 0.02 mg. are detectable. The range of acids covered is from C_1 to C_{12}, *i.e.* from formic to lauric acid.

The time at which a given acid emerges from the column is constant at a constant temperature and flow rate of nitrogen, so that the time of emergence can be used in the identification of an unknown acid. Furthermore, in any homologous series of compounds the energy of interaction with the stationary phase of the column increases by a constant amount for each increase of chain length by a CH_2 group, so that if the logarithm of the time of emergence of each homologue (or time of emergence relative to some standard substance) is plotted against the chain length of the molecule, a straight line results (Fig. 8) (see also Ray,[6] James and Martin[1]). In this way, from knowledge of the behaviour of a few members of a homologous series, the time of emergence of all other members of the series may be predicted.

The titration technique may be used equally well for volatile bases and Fig. 4 shows the separation of ammonia and the three methylamines completed in 35 min. The relative rates at which different substances move down the column are dependent on the nature of the forces (*e.g.* Van

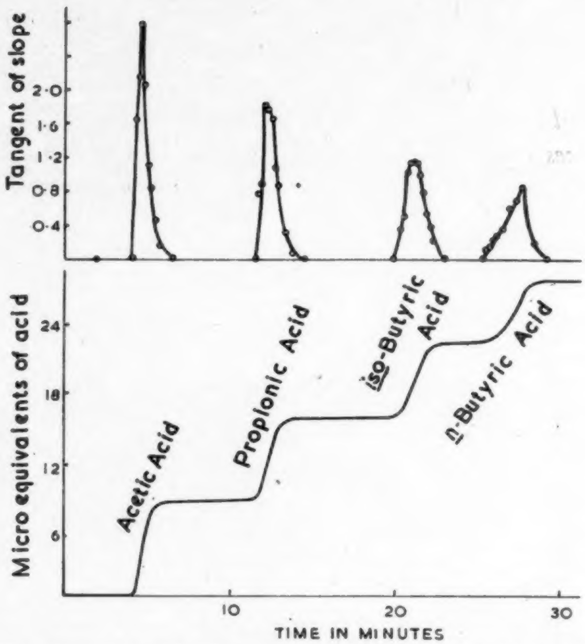


Fig. 2. The separation of acetic, propionic, *iso*-butyric and *n*-butyric acids at 100°C., using a 4-ft. column with a stationary phase of MS 550 silicone oil containing 10% w/w of stearic acid. Nitrogen pressure, 46 cm. mercury. Nitrogen flow rate, 45 ml. of nitrogen/min. Lower curve, experimental result. Upper curve, differential of experimental curve. Tangent of slope: 0.4, 0.8, 1.2, 1.6, 2.0.

Fig. 4. The separation of ammonia and the three methylamines using a 4-ft. column at 78.6°C. with a mixture of undecanol and liquid paraffin (15% v/v) as the stationary phase. Nitrogen pressure, 6.5 cm. mercury. Nitrogen flow rate, 5 ml./min. Lower curve, experimental result. Upper curve, differential of experimental curve. Tangent of slope: 1, 2, 3, 4.

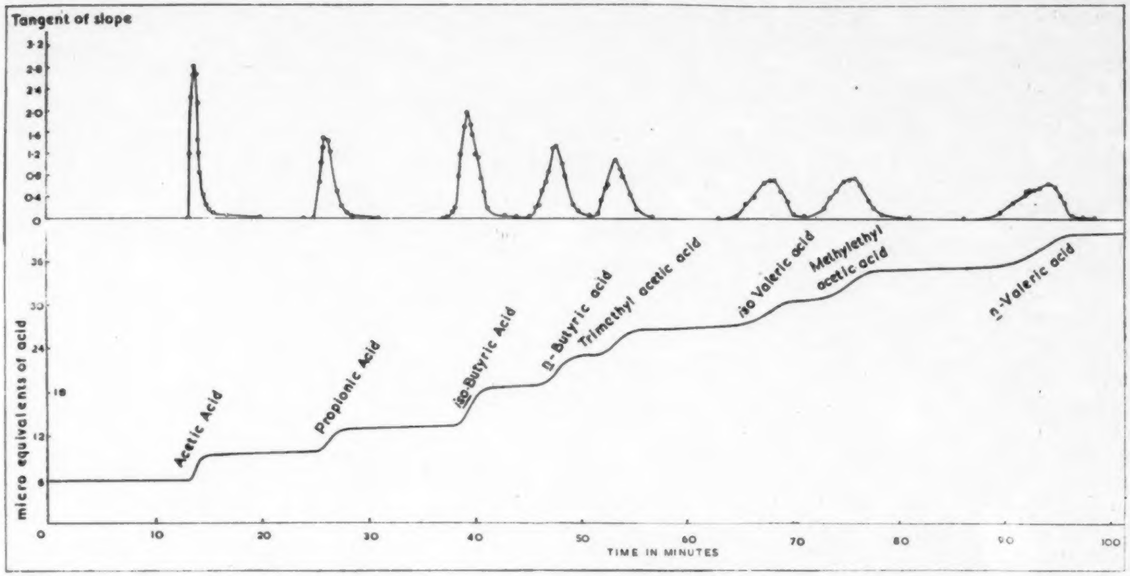


Fig. 3. The separation of acetic, propionic, *iso*-butyric, *n*-butyric acid and the isomers of valeric acid at 137°C., using an 11-ft. column with a stationary phase of MS silicone oil containing 10% (w/w) of stearic acid. Nitrogen pressure, 74 cm. mercury. Nitrogen flow rate, 18.2 ml./min. Lower curve, experimental result. Upper curve, differential of experimental curve.

der Waals forces, hydrogen bonding, etc.) involved in their solution in the stationary phase. For example, by variation of the chemical nature of the stationary phase the order of appearance of the three methylamines can be altered in any way desired. In the example shown the liquid in the column consists of a mixture of a long-chain alcohol and a long-chain hydrocarbon such that the best separation is achieved. If the stationary phase is changed to glycerol, then the order of appearance of the methylamines is reversed and a complete separation and quantitative determination of the three amines may be obtained in 8 min.[2]

In Fig. 5 is shown the separation of nine aliphatic amines on a 4-ft. column at 100°C. with liquid paraffin as the stationary phase. The order in which the amines appear in this case is determined only by the Van der Waals solution forces. Stationary phases such as long-chain alcohols or poly ethers alter the relative positions of the amines because additional solution forces due to hydrogen bonding come into play. If the relative positions of aliphatic amines on a liquid paraffin column (Van der Waals forces only) are plotted against their relative positions on a poly ether column (*e.g.* Lubrol MO, Imperial Chemical Industries) where hydrogen bonding can occur, it is found that the values obtained for primary, secondary, tertiary, dibasic and hydroxy amines fall on lines of different slope. In this way by running an unknown amine on these two types of column and measuring its time of emergence relative to a standard substance, the line along which the values fall will define closely the chemical structure of the unknown amine.[3]

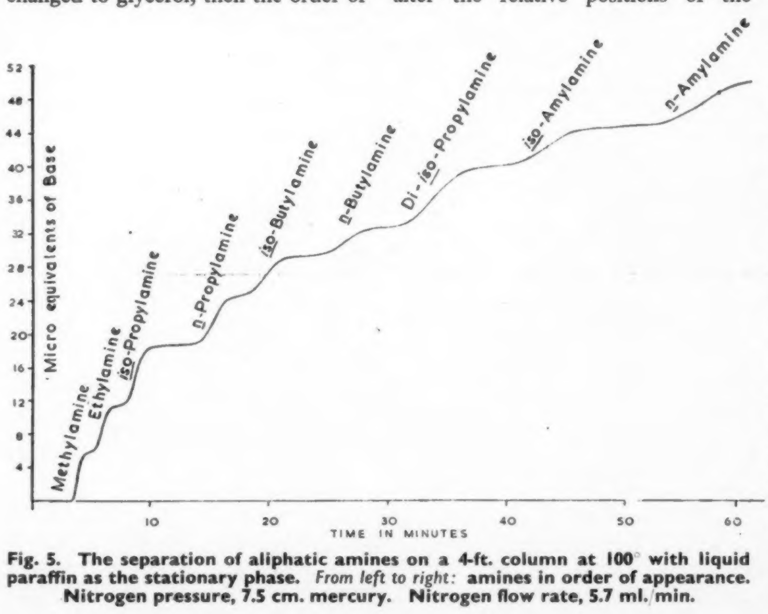


Fig. 5. The separation of aliphatic amines on a 4-ft. column at 100° with liquid paraffin as the stationary phase. *From left to right:* **amines in order of appearance. Nitrogen pressure, 7.5 cm. mercury. Nitrogen flow rate, 5.7 ml./min.**

Detection methods

In order to make the technique of gas-liquid chromatography applicable to all substances capable of being distilled, a more versatile method of detection than titration is required. Two techniques have been used for this purpose, the first involving the use of a catharometer. This instrument utilises the fact that the thermal conductivity of a gas is changed by the presence of small amounts of foreign substance. It consists of four channels containing fine platinum wires heated electrically and arranged to lie along the axis of the channels. When a gas passes down the channel the wire is cooled to an extent depending on the

thermal conductivity of the gas and the resistance of the wire changes. By using two comparator channels along which only the pure gas passes and by connecting the two running channels and the two comparator channels in a bridge circuit, it is possible to detect low concentrations of material emerging from the chromatogram. This type of instrument has been used by Ray[6] in the analysis of hydrocarbons, esters, ethers, etc., with gas-liquid chromatograms.

The second method takes advantage of the change in density of a gas stream caused by the presence of small amounts of volatile material. The instrument developed by James and Martin (unpublished) measures the difference between the density of the gaseous effluent from the chromatogram and that from a comparison column through which only nitrogen is passed, the two columns being maintained at the same temperature and with the same nitrogen pressure. The apparatus consists essentially of a series of tubes bored in a copper block (held at the same temperature as the columns) and connected in a manner analogous to a Wheatstone bridge so that a difference of flow rate of the two gas streams led into the block causes no pressure difference between two points in the system. Two sets of channels are connected by a cross-channel in such a way that any density difference between the two streams causes a pressure difference and hence a flow of gas through the cross-channel. This channel contains a flow detector consisting of a small filament (electrically heated) arranged below and equidistant from two connected thermojunctions; any cross-flow of gas causes the stream of hot convected gas from the filament to be diverted to one or other of the thermojunctions, heating one and cooling the other. The thermo-electromotive force produced is fed to a commercial d.c. amplifier whose output is led to a recording galvanometer. The galvanometer deflection is linearly related to the density difference of the two gas streams and, unlike the catharometer, is unaffected by changes in gas-flow rate.

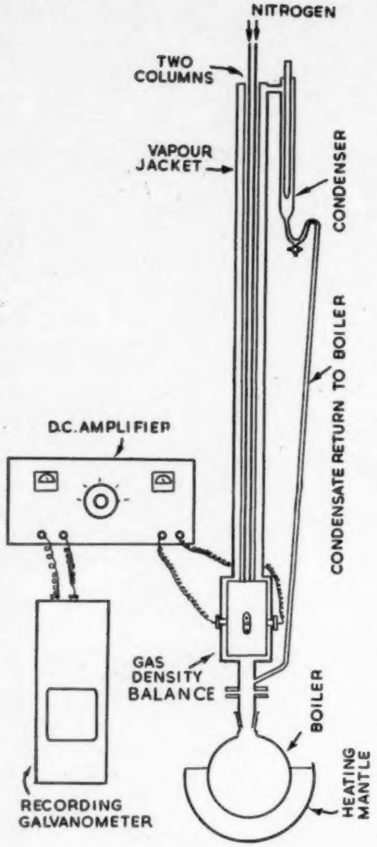


Fig. 6. Schematic layout of gas-liquid chromatogram using a gas density balance for detection and estimation of the material emerging from the column.

The instrument is highly sensitive, amounts of the level of $\frac{1}{10}$ μg. of amyl alcohol/ml. of nitrogen being detectable. A schematic diagram of the complete apparatus is shown in Fig. 6. As with the titration apparatus, the whole procedure is automatically controlled once the column has been loaded and the nitrogen turned on.

Some separations studied

The following diagrams illustrate a few of the separations studied. Fig. 7 shows a separation of the components present in commercial petroleum ethers using a column 11-ft. long at 78.6°. A similar if slightly better separation can be obtained by using a 4-ft. column at 23°C. with *n*-hexadecane as the stationary phase.

In Fig. 8 is shown the relationship between logarithm of the retention volume (or time of emergence × rate of flow of nitrogen), relative to *n*-pentane and the structure, of a variety of hydrocarbons from 2-methylbutane to *n*-decane for a column with paraffin used as stationary phase. All members of homologous series fall along lines of almost equal slope so that identification is often possible on the basis of time of emergence alone.

It has already been pointed out that useful information can be obtained by studying the behaviour of compounds of different chemical type on columns with stationary phases of different chemical structure where additional solution forces to Van der Waals forces may be involved. By the use of the graphical representation of behaviour on two types of column it is possible to distinguish between aliphatic,

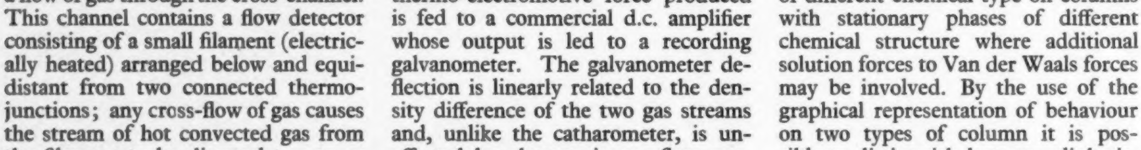


Fig. 7. Separation of the components present in commercial 'petroleum ethers' using a column 11-ft. long with a stationary phase of paraffin wax at 78.6°. Nitrogen pressure, 68 cm. mercury. Nitrogen flow rate, 16.3 ml./min.

Curve A: **'Petroleum ether,' boiling range 40 to 60°C. Load, 1.3 mg. Identification of peaks: (1) Air (introduced in loading the sample); (2) *iso*-pentane; (3) *n*-pentane; (4) 2:2 dimethylbutane (trace only); (5) *cyclo*pentane, 2-methylpentane and 2:3 dimethylbutane; (6) 3-methylpentane; (7) *n*-hexane; (8) methyl*cyclo*pentane, 2:2-dimethylpentane and/or 2:4 dimethylpentane.**

Curve B: **'Petroleum ether,' boiling range 60 to 80°. Load, 1.9 mg. Identification of peaks: (1) Air (introduced in loading the sample); (2) *n*-pentane; (3) *cyclo*pentane, 2-methylpentane and 2:3 dimethylbutane; (4) 3-methylpentane; (5) *n*-hexane; (6) methyl*cyclo*pentane and/or 2:2 dimethylpentane and/or 2:4 dimethylpentane; (7) 2:2:3 trimethylbutane; (8) *cyclo*hexane.**

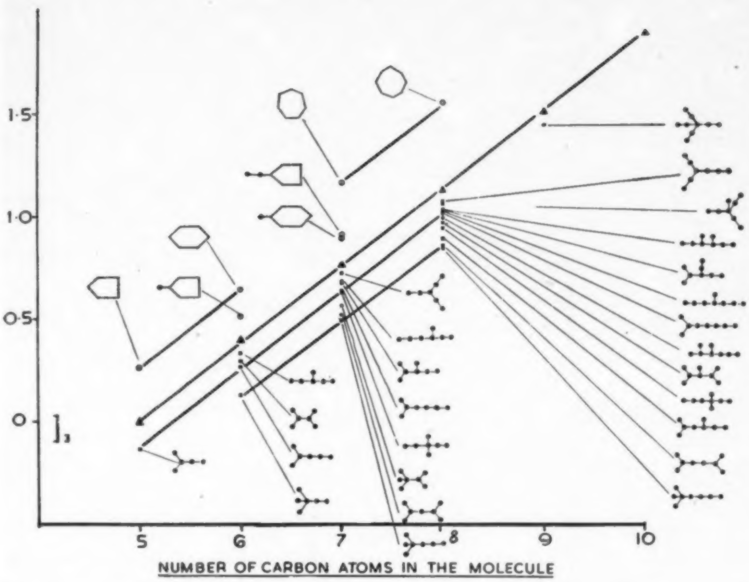


Fig. 8. The relationship between log retention volume relative to *n*-pentane (time for centre of peak to emerge relative to time for centre of *n*-pentane peak to emerge) in aliphatic hydrocarbons and the number of carbon atoms in the molecule, showing the effect of configuration on retention volume. The data are for a column with paraffin wax as the stationary phase at 78.6°C. The two vertical lines at the L.H.S. of the diagram show the vertical displacement necessary for complete separation and the minimum displacement necessary for a double peak to be shown.

naphthenic and aromatic hydrocarbons.

In Fig. 9 is shown the separation of aromatic hydrocarbons, where *m*- and *p*-xylene run so close together that they appear as a single peak. Increasing the length of the column (and hence its efficiency) would enable a separation of the two substances to be achieved. Fig. 10 shows the separation of the methyl esters of eight fatty acids completed in 90 min. In Fig. 11 is shown in curve *A* the separation of alkyl chlorides and in curve *B* the separation of a variety of ketones.

Table I. Some Separations Achieved

Substances	*References*
Volatile fatty acids C_1 to C_{12}	James & Martin[1]
Ammonia and the methylamines	James, Martin & Howard-Smith[2]
Aliphate amines and pyridine bases	James[3]
Aromatic bases	James[4]
Aliphatic hydrocarbons	James & Martin,[5] Ray,[6] Ray[7]
Aromatic hydrocarbons	James & Martin,[5] Ray[6]
Aliphatic alcohols	James & Martin,[5] Ray[6]
Aliphatic halides	James & Martin[5]
Esters	James & Martin,[5] Ray[6]
Ketones	James & Martin,[5] Ray[6]
Ethers	Ray[6]

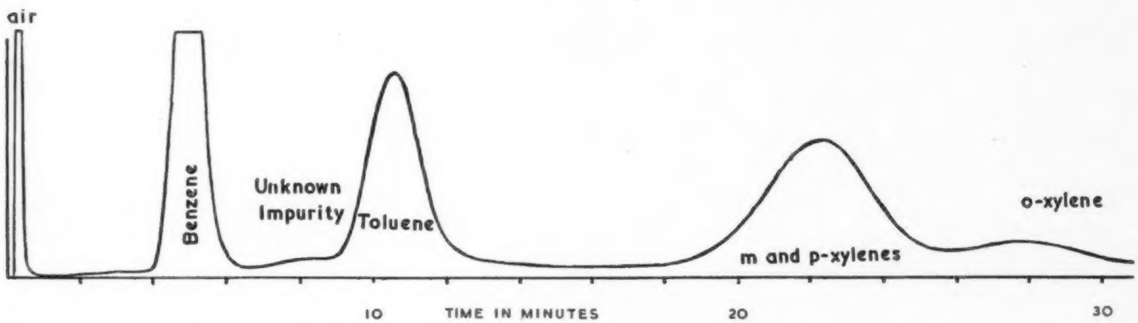


Fig. 9. The separation of aromatic hydrocarbons on a 4-ft. column with the aromatic hydrocarbon benzyldiphenyl as the stationary phase at 100°C. Nitrogen pressure, 42.6 cm. mercury; nitrogen flow rate, 42 ml./min.

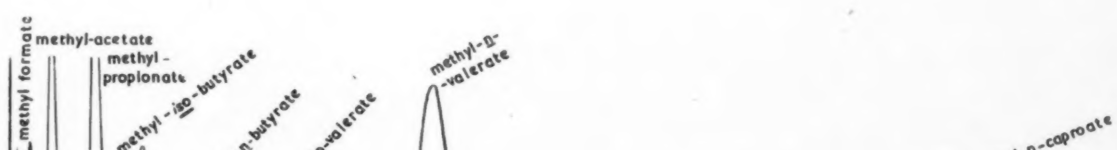


Fig. 10. The separation of a variety of methyl esters on a 4-ft. column having liquid paraffin as the stationary phase at 78.6°C. Nitrogen pressure, 27 cm. mercury. Nitrogen flow rate 20 ml./min.

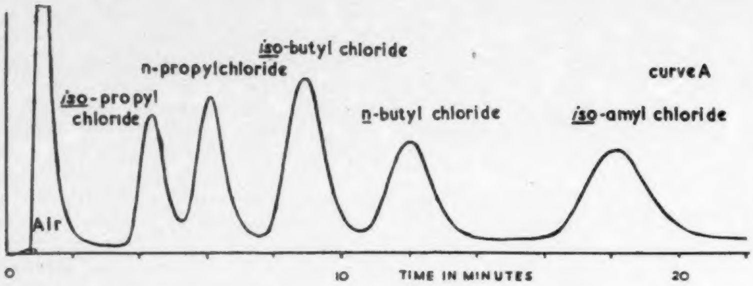
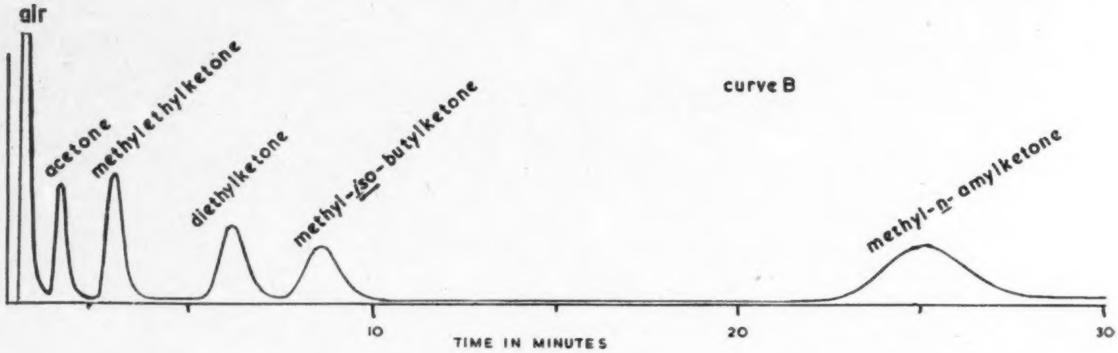


Fig. 11. The separation of alkyl chlorides and of ketones.

Curve A: **The separation of alkyl chlorides using a 4-ft. column with benzyldiphenyl as the stationary phase at 100°C. Nitrogen pressure 12.5 cm. mercury. Nitrogen flow rate, 11.8 ml./min.**

Curve B: **The separation of ketones using a 4-ft. column with paraffin wax as the stationary phase at 100°C. Nitrogen pressure, 17.8 cm. mercury. Nitrogen flow rate, 24.8 ml./min.**



The method should be applicable to all substances capable of being distilled and, by virtue of the high efficiency, high speed and relative simplicity, it should in time replace analytical distillation. In Table 1 is shown a list of substances run successfully on gas-liquid columns together with references.

There is no obvious reason why the method should not be scaled up to deal with much larger amounts of material, though at what level the apparatus becomes too cumbersome is impossible to say until a real study of this problem has been made. At this stage, however, columns dealing with 100 g. of material should be easily made. Fine-chemical manufacturers should find the method of great use even at its present stage of development, not only as an analytical tool of great versatility but also as a means of obtaining compounds of high purity from commercial sources on a scale suitable for laboratory supply.

Acknowledgments

I should like to thank the editors of the *Biochemical Journal* and the *British Medical Bulletin* for permission to reproduce diagrams and material already published.

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[2]A. T. James, A. J. P. Martin and G. Howard-Smith, *Ibid.*, 1952, **52**, 238.
[3]A. T. James, *Ibid.*, 1952, **52**, 242.
[4]*Idem* (largely unpublished, but see Ref. 5).
[5]A. T. James and A. J. P. Martin (details to be published, but see *Brit. Med. Bull.*, 1954, **10**, 170.
[6]N. H. Ray, *J. Applied Chem.*, 1954, **4**, 21.
[7]*Idem*, *Ibid.*, 1954, **4**, 82.

Dutch subsidiary for Dow

The Dow Chemical Co. has been authorised to establish a wholly owned subsidiary company in Rotterdam in the Netherlands, which will import, manufacture and distribute a basic line of chemicals for industries throughout the world. The new company, to be known as Nederlandsche Dow Maatschappij N.V., will process a variety of chemicals, plastics and magnesium alloys.

The new company plans to build a manufacturing plant as well as tank storage, warehouse and dock facilities on a 50-acre site leased for 20 years in the Third Petroleum Harbour at Rotterdam. The harbour is now being completed by the Port of Rotterdam, and construction on the plant site is expected to begin about mid-1955.

Among the products the firm plans to produce are *Styron*, Dow's polystyrene plastic; magnesium alloys; glycols including *Dowanols;* and other chemicals. Polystyrene production will include processing of *Styron* in a variety of colours for moulded applications.

Dow Chemical will use leased tankers to carry basic raw materials from its U.S. plants to the Rotterdam plant. The subsidiary also will purchase Dutch and other European raw materials. The Dutch firm will employ largely Dutch nationals, and certain qualified personnel will be sent to Dow plants in the United States for special training in production, technical advice and sales functions.

To Authors of Technical Articles and Books

The Editor welcomes practical articles and notes on chemical engineering and related subjects with a view to publication. A preliminary letter or synopsis outlining the subject should be sent to The Editor, CHEMICAL & PROCESS ENGINEERING, Stratford House, 9 Eden Street, London, N.W.1.

In addition, the Editors and Publishers of the Leonard Hill Technical Group are always ready to consider technical and scientific manuscripts with a view to publication. A synopsis with chapter headings should be sent in the first instance to the Book Production Manager, at the above address.

Plant and Equipment

Protecting threads at high temperature

A product recently introduced in the U.K. to solve the problem of seizing and galling of threaded connections subjected to prolonged exposure to extreme heat is *Thred-Gard*, a compound which, when painted on the surface of the bolt or stud before being fitted, protects the thread against this welding action. The makers of this compound, Crane Packing Ltd., describe it as a non-drip and non-hardening substance which lubricates and protects the threaded surface of studs, bolts, plugs, etc. Wrench torque is thus greatly reduced, and fittings can be drawn up to a greater degree of tightness. It is further stated that seizing and galling are prevented at operating temperatures up to 1,200°F., yet threaded connections can be easily disassembled.

Other applications for *Thred-Gard* include protection for thread-cutting dies and taps, broaching and spinning tools, wire-drawing dies, lathe centres, steady rests, rock drill pipe and bit threads. Cans are available in three sizes: ½ pint, 1 pint and 1 quart.

The underlying principle is the formation of a low-shear-strength metallic film upon the associated surfaces based on the constituent element, molybdenum disulphide. The makers state that this metallic constituent, when assisted by combination with the other ingredients, does in effect 'plate' the surfaces during the action of their contact, and the greater the specific loading the more pronounced is the affinity for attachment and consequent lowering of the coefficient of friction. This result is independent of temperature.

As an example, when a nut is tightened on a bolt against the resistance of loading, the lubricating effect of *Thred-Gard* is increased and thus the desired tension is achieved with far less effort, while the thread contact surfaces are prepared for subsequent dismantling without difficulty, or damage, through galling or seizing.

Hoses for H.T.P.

A range of thin-walled flexible hoses for handling H.T.P. (high-test hydrogen peroxide) has been developed by the Compoflex Co. Ltd. after intensive research carried out at the request of the Ministry of Supply. It is stated that the hoses are all entirely compatible with H.T.P. and have been approved by the various Government departments. A range of ancillary equipment, including O-rings and non-creep gaskets, is also produced for use with the hoses.

The hoses, which are claimed to be flexible over a wide range of temperatures, extremely hard-wearing, light and strong, are at present available in two types, each ranging in size from 1 to 3 in. internal diameter. A high-pressure hose is being developed.

One type of hose consists of a layer of specially compounded PVC known as *Molene*, covered with a layer of *Terylene* which has been coated on both sides with the same special PVC mix. Over this is wound an austenitic stainless-steel wire spiral covered with one layer of PVC and two layers of PVC-coated *Terylene*. The layers are then bonded together, under carefully controlled conditions of heat and pressure, to give a lightweight, smooth-bore flexible hose. The integral wire spiral allows the hose to support the weight of a man without damage. For 1½-in.-diam. hose, the weight per ft. is 0.87 lb. and the test pressure 200 p.s.i.

As an answer to the problem of designing a collapsible, ultra-light H.T.P. hose which could be wound on a reel, a sandwich-type hose, made up of a layer of specially compounded PVC between the two layers of PVC-coated *Terylene* has been produced. During manufacture a spiral channel is formed in the bore lining, and this rifling prevents the building up of gaseous products, as, no matter how much the hose is kinked, an escape passage is always provided along the entire length. Figures for the 1½-in. i.d. hose are: weight per ft., 0.25 lb.; test pressure, 150 p.s.i.

In the chemical and pharmaceutical industries, H.T.P. is used as an oxidising agent, in organic synthesis, in bleaching fats and oils, and soap.

The automatic control demonstration unit, described on this page. Here it is being examined by Sir Frank Whittle prior to his recent lecture at the Royal Institution.

Automatic control demonstration unit

The popular scientific lectures that are given annually at the Royal Institution in London were this year delivered by Sir Frank Whittle, consultant to one of the companies of the Royal Dutch/Shell Group, who took as his subject 'The Story of Petroleum.' It is almost a tradition that these lectures include particularly vivid practical demonstrations, and this year Foxboro-Yoxall Ltd. lent for the occasion an interesting exhibition unit designed to demonstrate the working of automatic control instruments such as might be found in a modern refinery. The unit is a transportable cabinet containing a number of instruments arranged to control the flow and level in a process simulated by a glass tank of coloured water. The front of the unit is a graphic panel with two Foxboro Model 53 *Consotrol*

recorders mounted in a representation of the plant. The unit can be swung round so that the other instruments with their connections and layout may be examined.

The flow, created by an electric pump, is measured at an orifice plate by a d/p cell transmitter whose measurements are recorded by one of the recorders. Here the desired value is set and control is effected by a Model 59 controller mounted on the head of a valve in the pipeline.

The level of the tank is measured by another d/p cell transmitter with an air-purged impulse dip leg and this measurement is recorded by the other recorder. As before, the desired value is set here and a Model 58 controller, this time mounted on the back of the recorder, controls a valve in the outlet pipe of the tank.

The demonstrator is able to create easily visible changes in the flow and level by varying the settings of the two Model 53 recorders and, as these instruments indicate both a departure from the desired value and the output pressure to the control valve, the response of the latter to changes in the process is easily appreciated. In short, the unit greatly facilitates the demonstration of the principles of automatic control to the non-technical besides permitting close examination of a number of typical modern control instruments while they are actually functioning as they would on a full-scale industrial process.

Liquid-level switch

A new type of flange-mounted, magnetic, liquid-level switch, the Type 1700, is designed for mounting on the side of tanks for the purpose of operating electric warning signals when the liquid either rises or falls to predetermined levels.

The mercury switch mechanism is operated by the rotation of a short float arm, the coupling between switch and float arm being made magnetically through the solid body of the instrument. It is claimed that this ensures complete absence of leakage under pressure tested to 80 p.s.i. and the casing is weatherproof.

The instrument may be supplied with the switch set either to make or break contact on changing levels, and the switching differential is $1\frac{1}{4}$ in. equally spaced about the centre line. The switch is of the mercury-to-mercury type, having a rating of 6 amp. at 250 v. a.c./d.c.

For traction and marine use a modified switch mechanism is employed which permits a special type of mercury switch to be tripped through 90°, thereby ensuring that the mercury will not leave the contacts under normal operating conditions. This model is usually supplied with a longer float arm and larger differential to prevent 'hunting' of the mechanism under conditions of liquid swirl.

Other models are available for use in high-pressure tanks, and modification can be made to suit corrosive conditions. The apparatus is manufactured in England by Bayham Ltd.

High vacuum rotary pumps

High vacuum is necessary for a large number of chemical uses such as serum tube filling, thermometer evacuating, disinfecting apparatus, distilling under reduced pressure and in the manufacture of drugs, chemicals, essential oils, insecticides, tooth powders and many other products. The latest design of the *Geryk* 8-in./50, single-stage-type, rotary, vacuum pump, a production of the Pulsometer Engineering Co. Ltd., is a compact, single-stage, water-jacketed pattern specially built for hard wear at low cost for factory use.

It is driven from any convenient source of power, *e.g.* an electric motor, on the same baseplates and vee-belts or by way of a flywheel pulley. The normal duty is 60 cu.ft./min. of free air or gas swept through, running at 350 r.p.m. taking not over 3 h.p., the vacuum obtained being 0.005 mm. off perfect on a closed dry circuit.

Automatic internal lubrication is provided so that all rotating parts run continuously in a film of oil, whilst the rotor has roller bearings, and the flywheel pulley runs on a trunnion drive, which takes all drive strain from the pump shaft and, the makers state, reduces gland trouble to a minimum.

When operating on condensable gas circuits, as in some sections of the chemical industry, a drip feed lubricator is provided, supplying clean oil continuously so that the internals of the pump are kept free of emulsified liquid. For circuits operating with moist air a special chamber is fitted on the discharge side in which any condensed water separates and prevents emulsification of the oil with no appreciable fall in the vacuum.

For certain classes of chemical work where it is not possible completely to trap and retain all corrosive and condensable vapours generated in the plant, this type of pump is available with protected rotor bearings.

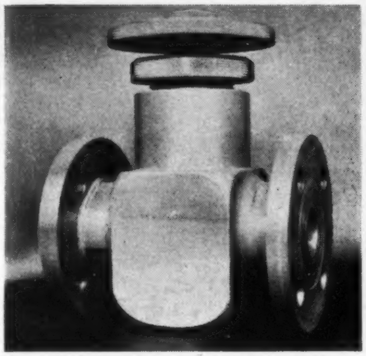
It is claimed that the arrangement prolongs the life between overhauls of the pump.

Membrane filters

A process for the production of membrane filters, which are now available in limited quantities, has been developed by Courtaulds Ltd. The filters are chiefly of use for the rapid bacteriological examination of water and sewage and for the filtration of pharmaceutical fluids or, in fact, for any work which requires the separation of bacteria or other fine particle suspensions from liquid or gaseous media. Membrane filters are stated to have considerable advantages over the conventional-type filters normally used for these applications in that much greater rates of flow through them can be obtained. In the cases where bacterial counts are required, the bacteria filtered off on the membranes can be subsequently grown by placing the filter in contact with liquid nutrients to give visible colonies on the surface of the filter. The used filters can afterwards be dried and kept as a permanent record. Other uses are in the preparation of bacteria- and dust-free water, the separation and subsequent assay of antibiotic drugs, the filtration of wines and soft drinks and the filtration of air for atmospheric pollution and pollen counts.

The filters are made of cellulose acetate and are thus soluble in certain organic esters and ketones, but they are unaffected by water, alcohol, ether and most hydrocarbons. The filters can be sterilised either by autoclaving at 10 to 15 p.s.i. pressure or by treatment with ethylene oxide.

The filters, which are about 0.005 in. thick, have a fine porous structure, the one surface consisting of a thin layer of very fine pores which are about 0.5 to 1μ in size, while the remainder of the filter has pore sizes of 3 to 5μ. It is stated that this type of structure enables suspensions to be filtered off on the surface of the filters and also permits a high porosity to be obtained. The membrane, although somewhat brittle when dry, becomes quite tough when wetted with water. The membranes can be stored indefinitely in a dry condition, without change of properties. The makers cite, as an example of the permeability of the films, flow rate at 70 cm. differential pressure of 80 cc./min./sq.cm. at a temperature of 20°C. It is possible in the production of these membranes to vary the size of the pores within certain limits if so desired. The films can be rendered transparent by immersion in white oil, thus enabling an easier microscopic examination of certain retained particles.



Rigid vinyl valve.

The filters are supplied in the form of discs marked with a $\frac{1}{4}$-in. grid for ease of making bacterial counts. At present, these discs are available up to 8 cm. diameter, but larger sizes will be available in the future. The membrane filters cost 6d. per 6-cm. disc for quantities of one gross or more.

Electronic detection of tramp metal

Extensive damage can be sustained by such equipment as crushers and attritors if tramp metal is carried into them with the raw material. Heavy-duty electronic equipment has been developed for the detection of tramp metal, including non-magnetic steels or non-ferrous metals, in all branches of mining, quarrying and mineral preparation, for the protection of primary and secondary crushers of all types, screens, washing plants, attritors, pulverisers and similar plant. Large pieces of tramp metal, such as digger teeth, crowbars, machine parts, fish-plates, etc., are stated to present no difficulty even where the moisture content is very high, while the smallest particle that can be detected depends on the type of material, the size of the material, the width of the conveyor and similar details appertaining to a particular plant. For instance, large particles may be detected in a rich ferrous ore, but small pieces could not be located owing to the effect of the ore itself necessitating a reduction in sensitivity. On the other hand, very small pieces can be detected in such materials as phosphate rock or limestone, as these have no effect on the detector.

A complete installation consists of two major items, the search coil unit and the control cabinet, together with the necessary interconnecting cables and certain auxiliary items. An oscillator in the control cabinet sets up a high-frequency electromagnetic field in the coil unit and, with no metal in the field, the system is adjusted mechanically and electrically to give zero signal. When a piece of metal passes through the search coil the field is disturbed, and the resultant signal amplified to operate a control relay, whose contacts may, in turn, control any desired external apparatus. In the simplest case, the relay may control the conveyor motor contactor, causing the belt to stop when metal is detected; in other cases it may control auxiliary apparatus to automatically deflect the metal to waste. In addition it may operate: (1) a signal lamp, situated remote from the control unit; (2) a warning horn or bell; and (3) a marking device to indicate the location of the metal on the belt.

If the system is arranged to remove the metal automatically, the detector can be arranged to reset automatically after a suitable time delay or, alternatively, it can be arranged for manual control by means of a push button resetting both the detector and the motor contactor for the main drive.

This equipment is supplied by Metal Detection Ltd.

Valves of solid rigid vinyl

The availability of *Cobex* rigid vinyl in block form has opened up great possibilities in the design and manufacture of valves. Hitherto, valve bodies were made from thin sheet pressed to the required shape and seam welded. This method had several disadvantages and valves manufactured in this way have been susceptible to mechanical damage. The repair of even small defects was liable to be expensive.

A new range of valves is machined from solid *Cobex* and intended for handling corrosive liquids. Excluding packing, the valve comprises five parts only: the body, spindle, gland nut, handwheel and handwheel nut. The flanges are screwed and welded to the body and generous proportions are allowed, giving ample strength. The spindle incorporates a special inlaid portion at the seating end which can be renewed when excessive wear has occurred. All the valves are tested to 80 p.s.i., but, it is stated, can withstand considerably higher pressures. This type of valve is at the moment available in sizes $\frac{1}{4}$ to 2 in. and it is hoped to increase this range in the very near future.

The valve is produced by Tanks & Linings Ltd., using *Cobex* rigid vinyl manufactured by BX Plastics Ltd.

Sizes range from $\frac{1}{8}$ in. (£1 17s. 6d. each) to 2 in. (£17 each).

NEW PRODUCTS IN BRIEF

New design of wire guard is being fitted as standard to Keith Blackman APA-type propeller fans. Makers state the guard makes accidental contact impossible, yet offers no great restrictions to air flow and does not create additional air noise. (Keith Blackman Ltd.)

★

Dry-chemical fire extinguisher, designed for speedier action in smothering fires involving petrols, oils, alcohols, paints and industrial solvents. Stated to be especially effective against 'running' fires, where flames are continually fed by flowing inflammable liquids. (Pyrene Co. Ltd.)

★

Silicone (class H) insulation is used in new range of 'dry'-type transformers. Makers state this class of insulation repels moisture and will not support combustion, and explosion hazards are reduced to a minimum. (Brentford Transformers Ltd.)

★

Highly concentrated, heavy, liquid, acid compound, *Lacron DL*, for rapid descaling of iron, steel and stainless steel. (Lacrinoid Products Ltd.)

★

Reinforced concrete walls for bulk storage, supplied in units 7 ft. high × 2 ft. wide. Two types are available—one designed to hold materials reposing at an angle of 35° at maximum loading pressure up to 50 lb./cu.ft., including coal coke, solid fuels, slag and ashes. Second type—also suitable for use as retaining walls and loading platforms—is intended for pressures up to 100 lb./cu.ft.: sand gravel, granite chips, lime, crushed stone, cement and minerals. (Marley Concrete Products.)

For further details of the plant and products described, please use the coupon on page 110

Chemical Engineering Invention

RECENT BRITISH PATENT CLAIMS

High-vacuum fractional distillation

A high-vacuum unobstructed path fractional distillation apparatus comprises a gas-tight evacuated column 12 having means, *e.g.* an electric coil 44, for heating its sides and a heated rotatable member at the lower end of the column with an upper vaporising surface 20. It also has means for feeding distilland on to the surface 20. A rotatable shaft 36 in the upper part of the column carries a brush, the outer ends of the bristles 34 of which touch the wall of the column. There is a condensing surface at or adjacent the upper end of the column.

In the apparatus of Fig. 1, distilland is fed on to the centre of rotatable surface 20, which is heated by electric element 24, by a conduit 26. Residue is taken off at 30. The condensing surface is a part of the wall of the column cooled by a coil 48 or air blast. Condensate collects in gutter 50. The shaft 36 is cooled by fluid forced through conduit 54. Vapours condensed on the shaft 36 are flung to the walls of the column by the bristles 34 and there revaporised, a portion of the vapours again condensing on the shaft 36. The alternate condensation and vaporisation with cross-current contact with vapours rising from surface 20 causes a tendency for the heavier components to pass downwards and the lighter components upwards.

In Fig. 2 distilland is fed to the centre of the column by line 26. Feed not vaporised on the walls of the column collects in gutter 64 and is fed by conduit 66 to vaporising surface 20. Residue from surface 20 is taken off by gutter 28 and may be recycled to feed line 26 by pump 70. The condenser 56 is separate from the column. The shaft 36 carrying the bristles 34 is cooled by air or water forced through conduit 54.—672,919, *Distillation Products Inc.*

FIG 2

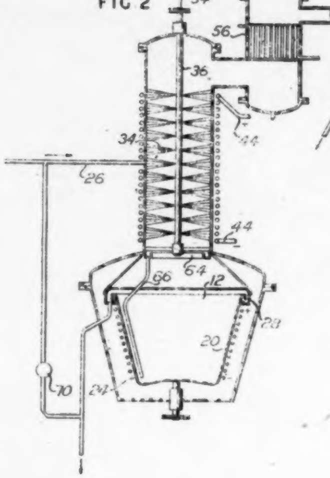
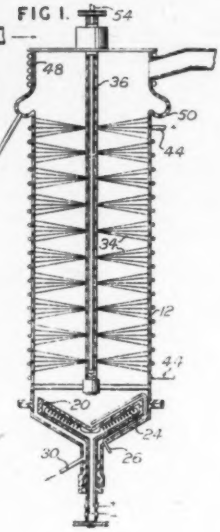


FIG 1.



Catalytic hydrogenation

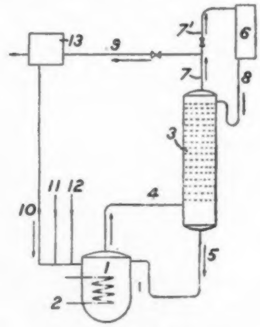
Organic compounds are hydrogenated in the presence of a Raney nickel catalyst at 20 to 180°C. using hydrogen containing 0.001 to 5% by volume of carbon monoxide. The catalyst is reactivated by being maintained at least periodically at 85 to 180°C., preferably in the presence of hydrogen. If the hydrogenation is carried out at 85 to 180°C., no separate reactivation step is necessary. The catalyst is preferably used in liquid suspension, for example as described in Specification 649,554 [*Group* IV (b)].

Unsaturated hydrocarbons, *e.g.* styrene or alkyl styrenes, unsaturated aldehydes and ketones, and saturated aldehydes and ketones, may be hydrogenated. Examples describe the preparation of isopropyl benzene from alpha-methyl styrene, butyraldehyde from crotonaldehyde, propionaldehyde from acrolein, 2-methyl-2: 4-pentane diol from diacetone alcohol, and methoxy-propanol from methoxy-propionaldehyde.—677,091, *Distillers Co. Ltd., T. Bewley and B. V. Aller.*

Lower aliphatic esters

Lower aliphatic esters are manufactured by continuously feeding the acid and alcohol to be combined into a heated aqueous reaction mixture containing an esterification catalyst, *e.g.* sulphuric acid, the water constituting a predominating proportion of the reaction mixture. The proportion of water in the reaction bath is preferably greater than 50% and it is advantageous for the bath to contain the alcohol in a lower proportion than that of the acid, preferably in a concentration of from 5 to 20%. The reaction is carried out in such a way that the concentration of the ester in the bath does not exceed 5%. The recovery of the evaporated reactants may be effected by known methods.

The vaporised acid may be recovered by submitting the vapours arising from the bath to washing by the liquid produced by the condensation of previous vapours in a proportion sufficient to constitute a barrage with respect to the acid vapours but not sufficiently



great to permit the formation of an azeotropic mixture of water and ester. The weight of the washing liquid is preferably between 0.2 and 0.5 times the weight of the vapour escaping from the bath.

In the drawing, an esterification kettle 1 has heating means 2, such as a submerged coil, and an escape conduit 4, for vapours, leading to the lower part of the plate column 3, at the foot of which is a return tube 5,

for liquids, communicating with the kettle 1. At the top of the column 3 is mounted a condenser 6 fed with vapours coming from the column 3 by a tube 7[1] fitted with a control cock; branching from the vapour take-off tube 7, 8 is a return tube for the condensed liquid. From the tube 7 a tube 9, provided with a cock, leads to a separating apparatus 13 in which the alcohol is separated from the ester. A tube 10 leads from the separator 13 to the kettle 1 and allows recovered alcohol to be returned thereto. Items 11 and 12 are tubes for leading in the acid and alcohol feeds. — 656,788, *Usines de Melle*.

Advances in Catalysis

IN this volume* the editors have maintained the high standard set by the earlier volumes in this series. The subjects treated range from the nature of the catalyst surface and the relation between catalytic constants and acid strength, to the chemical engineering aspects of catalytic cracking and isomerisation of hydrocarbons in the petroleum industry.

The section by J. T. Davies on 'Catalysis and Reaction Kinetics at Liquid Interfaces' gives an excellent review of the subject. The liquid interface is of particular interest as a site of catalytic activity because it is the simplest in conception and uniform over its whole area. It was suggested long ago that chemical equilibria at interfaces might be different from those in bulk phases. This has been shown to be the case and the causes for differences in reaction rates at interfaces as compared with the same reactions in bulk are now understood. Reactions at liquid interfaces have become of technical importance over the past few years and are exemplified by the production of synthetic rubber by emulsion polymerisation and the catalytic hydrolysis of fats in emulsions. A particular advantage of emulsion polymerisation is that the reaction rate and the molecular weight of the product can be separately controlled. It now seems well established that the polymerisation is initiated in the interior of the soap micelles and that these nuclei grow by attracting monomer from the bulk phase and from the droplets of monomer which are also present.

'Some General Aspects of Chemisorption and Catalysis' by Kwan deals with rates of chemisorption and equilibria on solid catalysts. The nature of the catalyst surface is discussed and the contribution which study of single crystals of metals in catalysis can make is reviewed. There still appears to be some dispute as to the role of discontinuities in the catalyst, even in the case of iron catalysts for ammonia synthesis. It is interesting to be reminded in the preface to this volume that Mittasch was largely responsible for the development of the iron oxide type of ammonia catalyst 40 years ago, and it is this type of catalyst which is still the basis for those used at the present day.

The section by Dunworth and Nord on 'Noble Metal—Synthetic Polymer Catalysts and Studies on the Mechanism of Their Action' is a useful guide to the possibilities of this type of catalyst. Colloidal metals of the platinum group were among the first catalysts employed in the hydrogenation of organic compounds. However, the disadvantages of these earlier catalysts are well known and the development in 1940 by Rampino and Nord of a new type of colloidal catalyst in which the metal is supported on a synthetic polymer such as polyvinyl alcohol has led to catalysts with a reproducible activity which may be used for a variety of organic solvents. The preparation, properties and composition of these catalysts are described, together with their applicability to various types of hydrogenation and the effect of water, oxygen and poisons on their activity.

'Interpretation of Measurements in Experimental Catalysis' by Weisz and Prater is a most interesting review of the possibilities of extracting the greatest possible amount of information from observations on the behaviour of catalysts. In catalyst research from the practical point of view, the most frequently encountered general problem is the determination of the so-called 'catalyst activity.' This catalyst activity is determined as a chemical reaction rate, but the measurements can be greatly influenced by gas transport phenomena and diffusion effects, which are quite unrelated to the fundamental properties of the catalyst. The article points out the pitfalls which can lead to misinterpretation of the results and describes sound methods for the determination of reaction velocity. The diffusion effects in experimental catalysis are considered under the headings of: Analysis of Diffusion Effects, Measurement of Intrinsic Reaction Velocities, Measurement of an Activation Energy, Real and Apparent Reaction Kinetics and Measurement of Catalyst Diffusivity.

'Commercial Isomerisation' by Evering is a general review of the development of the isomerisation of hydrocarbons for the production of high octane fuels for internal combustion engines. The processes described are applicable to the lower-molecular-weight hydrocarbons. Straight-chain paraffins have a very unfavourable performance in gasoline engines, while the branched-chain isomers have a much improved anti-knock performance. The early incentive was the production of isobutane from normal butane, for further conversion into iso-octane for aviation gasoline. The process can also be applied to the isomerisation of normal pentane and normal hexane, while a further interesting application was the conversion of methyl cyclopentane to cyclohexane and of dimethyl cyclopentane to methyl cyclohexane, which could be dehydrogenated to benzene and toluene in a subsequent process.

'Acidic and Basic Catalysis' by Kilpatrick reviews the various theories of acids and bases in relationship to the development of the theory of catalysts by acidic and basic catalysts.

For the chemical engineer, the section 'Industrial Catalytic Cracking' by Shankland will be of the greatest interest. This gives an excellent review of the catalytic cracking of petroleum hydrocarbons from the earliest developments in the 1930s up to the present time when fluidised bed catalytic cracking plants are processing about 500,000 tons of petroleum a day. The article covers the essential points of the fixed bed, moving bed and fluidised catalyst processes and about half the subject matter is devoted to the chemical engineering and constructional aspects. The composition of typical products such as gasoline and catalytically cracked gas oils is reviewed. The types of cracking catalyst employed commercially, the methods of manufacture of the catalyst and the desirable catalytic and physico-chemical properties are surveyed. The effect of different types of feedstock and methods of feedstock preparation on the performance of products of catalytic crackers is described and the article concludes with a good survey of the effects of the operating variables in typical catalytic cracking plants. The cover provided by this section is indicated by its length of 150 pages and 372 references.

P. J. GARNER.

**Advances in Catalysis*. Edited by Frankenburg, Komarewsky and Rideal. Vol. 6, Academic Press, 1954. Pp. 467, including index. $10.50.

World News

GREAT BRITAIN

Sulphuric acid plant contract

The Power-Gas Corporation Ltd. has recently been awarded a contract for the reconstruction of an existing sulphuric acid plant at the Flixborough works of Nitrogen Fertilisers Ltd. The new equipment will be built to the designs of Chemiebau Dr. A. Zieren G.m.b.H., and will comprise a converter unit with a capacity of 140 tons/day of monohydrate, an acid separator, waste heat boiler and ancillary equipment.

The programme for installation of the equipment has been carefully planned to reduce to a minimum the inconvenience of shutting down the existing plant.

Chair of coal science wanted

The lack of a chair of coal science at any British university was deplored by Dr. E. A. C. Chamberlain, chief scientist, and Mr. William Smith, deputy chairman of the Scottish Division of the N.C.B., in a paper to the Mining Institute of Scotland. In a country which owed its industrial structure to coal this omission was to be much regretted. Pioneer work has been done in Leeds, at the Imperial College, and at Sheffield and these, the speakers pointed out, were the only schools of fuel technology of university standing in Great Britain. They urged the foundation of such a chair in Scotland.

Finnish chemical engineer in Britain

A Finnish chemical engineer, Mr. Veikko Rauhala, is in the United Kingdom on a three months' British Council bursary to study the analysis of oil and oil products at Birmingham University's Department of Chemical Engineering. He is a research assistant in the peat and oil laboratory of the State Institute for Technical Research, Helsinki.

Record for British plastics

The British plastics industry had a record production and export year in 1954, it is announced by the British Plastics Federation. Output was more than 200,000 tons for the first time, about 25% more than in 1953. While most branches of the industry showed improvement, the advance was most marked in production of the thermoplastics materials—PVC (polyvinyl chloride), polythene and polystyrene.

Various expansion plans now under way will mean higher capacity still in 1955, and the biennial British Plastics Exhibition in London this year (June 1 to 11), it is expected will give another fillip to the export trade.

Exports in 1954 of plastics materials alone amounted to 70,000 tons, valued at more than £20 million, a rise of nearly £3½ million on the value for 1953, the previous highest figure. The tonnage is more than three times that for 1947 (21,000 tons) and more than 15 times that for 1938 (4,300 tons).

Australia, with an increase of more than £1 million to over £4½ million, remained the best market in 1954. Exports to India, New Zealand, South Africa, Holland, Denmark and Sweden, all showing substantial rises, were valued at between £1,200,000 and £1,400,000 each.

The exported materials consist of moulding powders, resins, sheet, rod, tube, film and foil, but do not include manufactured articles and parts.

Sulphuric acid and oleum

The summary of monthly returns issued by the National Sulphuric Acid Association Ltd. for the year 1954 shows a production of 634,363 tons (chamber and tower) and 1,408,129 tons (contact), making a total of 2,042,592 tons.

'Electricity and the Future'

Under the above title, the seventh British Electrical Power Convention is to be held at Brighton from June 27 to July 1, 1955. Associated with the convention will be the largest and most comprehensive electrical exhibition ever staged outside London, with the possible exception of the B.I.F. at Birmingham. Among the papers read will be one on 'The Use of Electricity in the Chemical Industry,' by Mr. G. Nonhebel, B.A., B.SC., F.R.I.C., F.INST.F., head of the fuel economy section of Imperial Chemical Industries Ltd.

Instruments exhibition

The third British Instrument Industries' Exhibition to be held at Earls Court, London, from June 28 to July 9, 1955, will show the latest equipment of manufacturers of scientific and electrical instruments, industrial meters, automatic controls, scientific glassware, drawing office equipment, and their associated manufacturers.

Oil refinery modernisation

J. O. Buchanan & Co. Ltd., of Renfrew, have now completed the modernisation and reconstruction of their oil refinery at Renfrew after fire damage about two years ago. The firm claims to be the oldest and only lubricating oil refinery in Scotland and has a range of some 500 products from the lightest types of spindle oils to the heaviest greases.

In modernising and rebuilding, the opportunity has been taken of extending the productive capacity and the range. Plant now being installed will provide for production of copper naphthenate, used in anti-rot practice in the heavy textile industry.

Akers research laboratories

Imperial Chemical Industries Ltd. are renaming their Butterwick research laboratories, located at the Frythe, Welwyn, Herts., in memory of the late Sir Wallace Akers, C.B.E., F.R.S. These laboratories were formed shortly after the end of the war to undertake fundamental and long-distance research work in various branches of science. Sir Wallace, in his capacity of I.C.I. research director, took a keen personal interest in the project, and was closely associated with the establishment of what, in industry, is a rather novel type of research activity.

Sulphur Exploration Syndicate

The British Sulphur Corporation Ltd. has assumed the functions of the executive office of the Sulphur Exploration Syndicate. The change is made partly for administrative reasons and partly to provide the necessary financial backing for a continuance of the activities of the Sulphur Exploration Syndicate, under the same management. The offices of the Corporation are at 4 Grafton Street, London, W.1.

The objects of the Corporation are similar to those of the Syndicate, though there is no longer the same need to emphasise the exploratory activities. In order to keep abreast of new developments, the Corporation will maintain its interest in new sulphur-producing projects and processes which, when desirable, would be publicised in a quarterly bulletin. In appropriate cases, the Corporation may, either directly or indirectly, consider financial participation in a sulphur-producing project or extending assistance in the disposal of sulphur so arising.

Protecting structural steel

The Society of Chemical Industry (Corrosion Group) is holding a symposium on the 'Protection of Struc-

★ PERSONAL PARAGRAPHS ★

★ **Sir Ewart Smith** has been elected a deputy chairman of Imperial Chemical Industries Ltd. (The company has two other deputy chairmen—Mr. A. J. Quig and Mr. S. P. Chambers, C.B., C.I.E.) He joined Synthetic Ammonia & Nitrates Ltd., forerunner of I.C.I.'s Billingham Division, in 1923 and for 19 years served at Billingham in various engineering and managerial posts. In 1932 he was appointed chief engineer of the Billingham Division. From 1942-45 he was chief engineer and superintendent of armament design at the Ministry of Supply. He was appointed to the board of I.C.I. in 1945, becoming technical director in 1946. He is chairman of the British Productivity Council.

★ **Mr. H. H. Smith,** who has been assistant manager of the special vessels department of Babcock & Wilcox Ltd. since 1950, has been appointed sales manager of Edwin Danks & Co. (Oldbury) Ltd.

★ **Dr. H. M. Stanley** has been appointed controller of research and development of The Distillers Co. Ltd. in succession to **Dr. F. Roffey,** who remains technical director of the company and has taken on the additional duties of personnel director of the company's industrial group. Dr. Stanley will shortly complete 25 years of research with the company. He became deputy controller of D.C.L. research and development in 1945. A graduate of Birmingham University, where he worked under the late Prof. G. T. Morgan, he is known particularly for his investigation into the production of petroleum derivatives and the economics of their application in the chemical and plastics fields.

★ **Col. G. W. Raby,** C.B.E., has been appointed Deputy Director (Engineering) to the Research Group of the United Kingdom Atomic Energy Authority at Harwell. He will take up his appointment in April. He has had a distinguished career as an engineer both in private industry and Government service. Since 1951 he has been chairman and managing director of the Sudan Gezira board.

★ **Mr. O. D. Gwilliam** has been appointed works manager for W. C. Holmes & Co. Ltd.

★ We announce with regret the sudden death of **Mr. D. Goldberg,** sales manager of Keith Blackman Ltd., who was with the company for over 38 years.

★ As a consequence of the above, Keith Blackman Ltd. announce the following appointments: sales manager, **Mr. C. J. Atkins;** assistant sales manager, **Mr. A. H. Woodley;** contracts manager, **Mr. F. W. Goodge;** assistant to the chief engineer, **Mr. D. Humphrey.**

★ **Dr. George W. Rigby,** of Wilmington, Delaware, U.S.A., has been appointed European technical representative of E. I. du Pont de Nemours & Co.

★ Monsanto Chemicals Ltd. announce the appointment of **Mr. N. F. Patterson,** director and general manager of production, as operations director responsible for all manufacturing operations of the company.

★ **Mr. T. E. Laing** has been appointed a director of British Resin Products Ltd. Mr. Laing, who joined the D.C.L. group in 1943, has been works manager and more recently general manager of the joint British Resin Products Ltd./British Geon Ltd. factories at Barry, Glamorgan, since they were built from 1947 onward. He began his career as a chemical engineer with the Anglo-Iranian Oil Co. Ltd. in 1922. He later served with the Socony-Vacuum Group as operating manager in Britain and in Germany and Italy.

★ **Mr. C. Paine,** chairman of the Dyestuffs Division, and **Mr. W. D. Scott,** a managing director of the Billingham Division, have been appointed to the board of Imperial Chemical Industries Ltd. as executive directors. Mr. Paine has been chairman of the Dyestuffs Division since November 1952. Mr. Scott worked for some years with the Hercules Powder Co. Ltd., in America and Holland. In July 1935 he joined I.C.I. in the Explosives Division sales department and six years later was transferred to the Dyestuffs Division as market development officer, later becoming homes sales manager for the division. In 1951 he went to the Billingham Division, being appointed joint managing director (commercial) in the following year.

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tural Steel' on March 31 and April 1. It will be held at the Institution of Civil Engineers, London. Non-members of the Society are cordially invited to attend and take part in the discussion, provided that they register beforehand and pay the conference fee of £2.

British glassware for Germany

An order for large-scale evaporation equipment for the pharmaceutical industry has been received by Q.V.F. Ltd. from a German firm. From 1900 to 1939, Germany was preeminent in the laboratory and industrial glassware industry, and it is thought that this will be the first British industrial glassware to go to Germany. It is believed that there is every prospect of an extension of British business within Germany. British glassware exports to Europe and Scandinavia are also progressing. Q.V.F. Ltd. state that, at the present time, competition from Germany is quite limited.

Another order received by Q.V.F. recently is for two 40-ft.-high, 18-in.-diam. glass fractionating towers for the Dutch chemical industry.

Instruments on show

Indicating and recording instruments, fractional-h.p. motors and the *Noflote* control for drainage and other pumps will be included in the exhibits of Evershed & Vignoles Ltd. at the Electrical Engineers (A.S.E.E.) Exhibition at Earls Court from March 15 to 19. Numerous testing instruments will also be shown by the company, while among the smaller instruments is a dual-scale thickness gauge for measuring the thickness of non-magnetic coatings such as paints or plating on ferrous bases. Evershed cable cleats will be shown for the first time.

INDIA

More industrial projects

Although the Government of India's first five-year plan does not end until the middle of 1956, consideration is already being given to the framing of the second five-year plan, in which, it is understood, greater emphasis will be placed on industrial development.

Proposals for the setting up of various new industries and the building of a number of new plants have been submitted by State Governments to the Central Government. Among the products that would be involved are aluminium, paper, insecticides, soda ash, soap, ferro-manganese, fertilisers, sugar, towns gas, tungsten, lignite, cement and sulphuric acid (from gypsum), calcium carbide and numerous others.

Since the State Governments do not command technical facilities to

the same extent as the Central Government does, and most of them do not possess sufficient resources of their own for carrying through such projects to completion and are not well organised for running them, it is expected that large-scale industrial developments in the public sector will be taken up by the Government of India themselves, either directly or through the National Industrial Development Corporation.

CANADA

Sulphur from natural gas

As a result of extensions costing $500,000 to Shell's plant for the extraction of sulphur from natural gas at Jumping Pound, Alberta, production of sulphur will be increased to 80 tons/day, almost trebling former capacity. Shell has thus increased its sulphur-extraction capacity at Jumping Pound to match the recent $1½-million expansion of its natural gas facilities in that area. Since last November the enlarged plant has been producing 60 million cu.ft./day for town lighting and domestic purposes. Its previous daily output was 35 million cu. ft.

NORWAY

Norsk Hydro's surplus

The Norsk Hydro chemical concern had a trading surplus of £6,450,000 in the 1953-54 financial year. In its annual report the company states that since 1945 it has invested altogether £25,850,000 in new plant. All except about £3 million was financed from the company's own resources. The company produces chiefly nitrogenous fertilisers, but also manufactures a great variety of other chemical products. One of Norsk Hydro's products is heavy water.

Big steel plant to open soon

The opening of the A/S Norsk Jernverk's steel plant at Mo i Rana, northern Norway, this year, will be a most important step in the development of Norwegian industry. The plant, built as a result of a Parliamentary decision in 1946, is the first large integrated steel mill in the country. Initially, output will be some 170,000 tons p.a. of rolled products. When a fourth pig-iron furnace has been installed, capacity will be around 230,000 tons and it is hoped eventually to raise production to 500,000 tons. Norway now has to import some 400,000 tons p.a. of steel.

The country has very large iron-ore deposits, production from which has until now mostly been exported. At the outset, ore for Mo i Rana will come from the Fosdalen and Syd Varanger mines, but in due course it is planned to exploit the very rich deposits in the Dunderland Valley, a few miles from Mo.

DENMARK

Export restrictions eased

The Danish authorities have removed the export ban from tar, vegetable oils and certain other products, while items on which restrictions have been eased include several types of machinery, electrical equipment, chemicals, metals and minerals, mineral oil products, rubber and rubber products.

At the same time, the Government has cancelled a ruling that free exports of goods supervised by the Ministry of Commerce, to countries belonging to the European Payments Union, must be paid for under mutual payments agreements or in dollars.

TURKEY

Industrial projects

Among several new factories to be established in the Adana area is a fertiliser plant which is to produce enough to allow for considerable exports to Arab countries.

Other projects envisage the building of a sugar refinery to process cane sugar, a paper factory and a cement plant.

PHILIPPINES

Copper plant

Atlas Consolidated Mining & Development Corp., of Toledo, on the island of Cebu, is opening a 200-ton/day copper concentrating plant. The plant will draw its raw material from reserves estimated to contain 35 million tons of ore averaging 1.05% copper. It will also have an estimated daily output of 250 tons of pyrites and $2,000 worth of gold.

SPAIN

Chemical output raised

In 1954, Spanish output of mineral potash (in terms of K_2O) rose by 12½% to 182,000 tons, according to a survey by the Minister of Industry. Pyrites production advanced by 6% to 1,894,000 tons, and sulphur by 3% to 41,600 tons. In October a new factory for nitrogen fertilisers, with a capacity of 50,000 tons p.a., started production in Barros, Asturias.

Output of Spain's heavy chemical industry was as follows (in metric tons): nitrogen fertilisers, 160,000; superphosphates, 1,417,000; sulphuric acid, 744,000; sodium carbonate, 102,000; caustic soda, 91,000; rayon-making pulp, 16,500; paper-making pulp, 149,000.

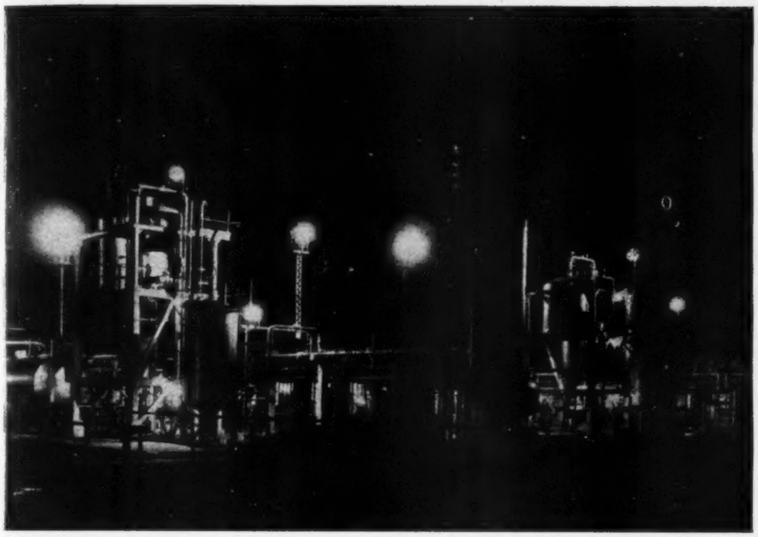
Glycerine and sodium nitrate

Imports of glycerine into Spain from sources other than Holland, from where imports have recently been arriving, are likely to be allowed by the Ministry of Commerce. It is also reported that 50,000 tons of sodium nitrate will shortly be imported from the Sociedad Commercial de Nitrate de Chile.

WESTERN GERMANY

I.G. Farben successor

The West German Liquidation Committee of the former I.G. Farben chemical combine has announced the formation of the Chemie Verwaltungs A.G. as a holding company for the Chemische Werke Huels A.G. with a capital of 81,600,000 marks. Huels, formerly part of I.G. Farben, is at present the sole producer of synthetic rubber (buna) in West Germany. The



An important centre of chemical engineering industry in Scotland is the Grangemouth oil refinery of the British Petroleum Co. This recent night view shows the copper chloride units. No. 1 (*foreground*) sweetens straight-run benzine and No. 2 (*background*) sweetens catalytically-cracked spirit.

shares of the holding company will not be distributed, but will be placed under trusteeship until all claims against the former I.G. Farben company have been settled, states the Committee.

ISRAEL

Gypsum plant

A new gypsum plant has started production in the Wadi Ramon in the Central Negev, built by Even Ve Sid Ltd. and the settlement of Sde Boker. The Even Ve Sid Ltd. is a subsidiary of Solel Boneh. The plant is expected to produce 150 tons/day of crushed gypsum and the first shipment has already been despatched to the Nesher cement factory at Haifa.

JAPAN

Fertilisers and antibiotics for China

China will send Japan rice, soya beans, salt and other commodities in exchange for chemical fertilisers, penicillin and other medicinal products. Contracts have been signed for about £10 million's worth of goods.

FRANCE

Chemicals share in industrial recovery

Provisional figures issued for 1954 show an 8% rise in industrial production. Chemicals (15% rise) are among industries in which recovery has been particularly notable. This is not the case with leather and fats and oils, production of which is still below the pre-war level.

Under pressure from the Government and the Charbonnages de France, the oil industry has agreed to limit production of fuel oil in 1955 to only slightly above the 1954 level. This is in addition to accepting a 400-francs tax on fuel oil. The Charbonnages de France have agreed to a 3% reduction in the price of industrial coal for use in factories.

Chemical pulp plant

A plant to produce 40,000 tons p.a. of chemical pulp from hardwood, the first of its kind in France, has started operation. It is owned by the Société Industrielle de Cellulose d'Alizay.

Consumption of pulp by the French textile industry totals 120,000 tons p.a. and, because of the inadequate quantities of softwood available in France, 10,000 tons of timber has had to be imported p.a. for pulp production. The new plant will thus save foreign exchange equivalent to 2,500 million francs p.a.

SOUTH AFRICA

Chlorine and caustic soda expansion

African Explosives & Chemical Industries Ltd. anticipate an increasing demand for chlorine and caustic soda, and to cope with this they have cleared 20 acres of land at the Umbogintwini factory, near Durban, for new plant and the roads and railways to serve it. Initially the new plant has a floor space of 137,000 sq. ft., but it has been designed for rapid expansion as the future demand increases. About £2 million is being spent on this section, with much of the plant made locally, including the pressure vessels, which previously were all imported.

In the beginning it is expected that a quantity of the salt needed in the manufacture of chlorine may be imported, as salt of sufficiently high grade may not be available locally, but it is hoped that eventually all these needs will be supplied in the Union.

The caustic soda produced in the new plant is to be sold as 45% liquor, some being reacted with chlorine to form a solution of sodium hypochlorite, which is needed as a raw material by a number of local industries. It is expected that several thousand tons of hypochlorite liquor will be obtained annually.

A separate section has been designed to burn hydrogen and chlorine to yield hydrogen chloride, this gas being absorbed in water in glass towers to produce up to 1,000 tons p.a. of hydrochloric for sale to industry.

In the chlorinated solvents section, inflammable acetylene and acrid chlorine will be used to form trichlorethylene and perchlorethylene. African Explosives & Chemical Industries report an increasing demand in southern Africa for polyvinyl chloride and it will be the first local undertaking to produce it from its basic raw materials. With the growth in South Africa of electric cable, hose, leathercloth and other plastic industries, there is now a big demand for PVC.

All sections of the new plant are expected to be in full commission by the middle of 1955, but peak production may not be reached until the end of this year. Much of this expansion work has been underaken with the technical assistance of experts from the British organisation closely associated with the South African company.

UNITED STATES

American Cyanamid's thermoplastics projects

Plans for the entry of American Cyanamid Co.'s plastics and resins division into the broad field of thermoplastics were announced recently. Ground has been broken at the company's Wallingford, Connecticut, plastics plant for construction of a new unit to produce some 40 million lb. of thermoplastics moulding compounds. At the same time, construction of a new unit is being started at the company's Fortier plant near New Orleans, Louisiana, to produce a new intermediate as a raw material for the Wallingford operation.

The processes for producing the

moulding compounds as well as the principal intermediate, monomethyl styrene, have resulted from several years of research in the company's research laboratories at Stamford, Connecticut. These processes are new and patented. It is believed they will not only result in new polymers and copolymers of interest in the field of plastics, but also new intermediates that may be useful to other divisions of the company.

Pharmaceutical projects

Chas. Pfizer & Co. Inc., the American pharmaceutical firm whose £2½-million production and refining project on the Kent, England, coast is nearing completion, announces it is carrying out a simultaneous expansion programme on the other side of the world. A new plant for producing the newest antibiotic drugs, terramycin and tetracyn, is being prepared on the outskirts of Manila in the Philippines, and is due to start operating in the early spring. Other plans for the Far East include a long-term programme for Korea.

AUSTRALIA

Kwinana refinery starts up

The British Petroleum Co.'s new 3-million-tons-p.a. Kwinana refinery, near Fremantle, Western Australia, started up recently. Kwinana is now the largest oil refinery in operation in Australasia. Built at a cost of about £30 million in 27 months, it has begun to operate more than three months ahead of schedule. The refinery will be operated by Australasian Petroleum Refinery Ltd., one of B.P.'s Australian associates. Products from the refinery will include petrol, kerosene, and diesel and fuel oils.

The main refinery plant consists of two distillation units, each with a capacity of 1½ million tons p.a. of crude oil, a catalytic cracker, a platformer and a hydrofiner. Overall contractors were the Kellogg International Corporation and construction was carried out by a consortium of United Kingdom companies, comprising Costain-John Brown Ltd., Kinnear Moodie Ltd. and D.C. & William Press Ltd.

In addition to the refinery, an oil port has been built which is capable of accommodating up to three of the company's largest tankers (32,000 d.w. tons) at one time. At the peak of the construction period, 3,500 workers, both skilled and unskilled, were employed. When all the units are in operation, Kwinana will be operated by a staff of about 1,000. To house this staff, a complete township with all the necessary amenities has been built by the Western Australian State authorities.

MEETINGS

Institution of Chemical Engineers

March 12. ' Pilot Plant and Scale-up Problems,' 2.30 p.m., College of Technology, Manchester.

March 15. ' Correlations for use in Transport of Aqueous Suspensions of Fine Solids through Pipes,' by K. E. Spells; ' Experiments on the Flow of Sand-Water Slurries in Horizontal Pipes,' by R. A. Smith; ' Hydraulic Conveying,' by C. J. Richardson, 5.30 p.m., Geological Society, Burlington House, London, W.1. Joint meeting with Chemical Engineering Group, Society of Chemical Industry.

March 16. ' Instrumentation of a Nuclear Power Plant,' by P. V. Koller, 7 p.m., College of Technology, Manchester.

Society of Chemical Industry

Chemical Engineering Group

March 25. ' Chemical Engineering Developments in the Coal Industry,' by G. I. Jenkins, at Swansea.

Road and Building Materials Group

March 17. ' The Practical Implications of Fundamental Research on Cement Hydration,' by Dr. J. W. Jeffery, 6 p.m., Institution of Structural Engineers, 11 Upper Belgrave Street, London, S.W.1.

Institute of Fuel

March 18. 'Application of Gas Turbines to Industry,' 7.15 p.m., Loughborough College, Loughboro'.

March 22. 'Advances in Complete Gasification,' by Dr. F. J. Dent, 7 p.m., M.E.B. Lecture Hall, Kingsway, Stoke-on-Trent.

April 20. Visit to laboratories of Chemical Engineering Department, Birmingham University; ' Training of the Chemical Engineer in Fuel Technology,' by Prof. F. H. Garner and Dr. R. Long.

Manchester Association of Engineers

March 25. Annual General Meeting and ' Engineering Problems Associated with Production and Operations at Low Temperature,' by H. E. Charlton, 6.45 p.m., Engineers' Club, Albert Square, Manchester.

Institution of Mechanical Engineers

March 25. ' Hydraulic Transport of Solids,' by R. C. Worster and D. F. Denny, 5.30 p.m., 1 Birdcage Walk, Westminster, London, S.W.1.

Junior Institution of Engineers

March 18. ' Some Notes on the Flow of Fluids,' by R. F. Twist, 7 p.m., Pepys House, 14 Rochester Row, London, S.W.1.

INTERNATIONAL MEETINGS

March 20-23. Meeting of the American Institute of Chemical Engineers at Houston, Texas.

March 20-23. Symposium on ' Heat Transfer,' American Institute of Chemical Engineers, Louisville, Kentucky.

March 21-25. Conference on ' The Functions and Education of Chemical Engineers in Europe ' (E.P.A. Project No. 297). Information from D.S.I.R. or Inst.Chem.E., London.

March 24-26. Annual meeting of Swedish Paper and Cellulose Engineers, Stockholm.

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